REMARKS

Applicants appreciate the Examiner's thorough review of the present application, and respectfully request reconsideration in light of the preceding amendments and the following remarks.

Claims 1-19 are pending in the application. The original claims have been amended only to improve the claim language. Claims 15-19 have been added to provide Applicants with the scope of protection to which they are believed entitled. No new matter has been introduced through the foregoing amendments.

The specification has been revised and Figs. 6-7 have been added to include the material previously incorporated by reference, i.e. the Japanese standards JIS-L-1092 and JIS-Z-0208 mentioned in page 7 of the originally filed specification.

More particularly, the revised specification (Substitute Specification) now includes the descriptions of

- The low hydraulic pressure method (Method A) for determination of the water resistance of textiles, which is found in pages 5-6 of JIS-L-1092; and
- The Dish Method for determination of the water vapour transmission rate of moisture-proof packaging materials, which is found in pages 1-5 of JIS-Z-0208.

Complete English translations of JIS-L-1092 and JIS-Z-0208 are enclosed as Exhibits A and, respectively, for the Examiner's reference. A person of ordinary skill in the art, based on the provided complete English translations of JIS-L-1092 and JIS-Z-0208 would recognize that the amendatory material does not introduce new matter into the disclosure of the present invention. A declaration is also attached, pursuant to the decisions of *In re Hawkins*, 486 F.2d 569, 179 USPQ

157 (CCPA 1973); *In re Hawkins*, 486 F.2d 579, 179 USPQ 163 (CCPA 1973); *In re Hawkins*, 486 F.2d 577, 179 USPQ 167 (CCPA 1973). *See* also *MPEP*, section 608. 01(p).

Entry of the Substitute Specification and approval of new Figs. 6-7 are believed appropriate and therefore courteously solicited.

The *35 U.S.C. 112, second paragraph* rejection of claim 1 is believed overcome in view of the amendments made to claim 1. The *35 U.S.C. 112, second paragraph* rejection of claim 11 is believed overcome in view of the changes made to the specification and new Figs. 6-7.

The *35 U.S.C. 102(b)* rejection of claims 1-4, 7-10 and 14 as being anticipated by WO 96/38620 (PCT/US96/07938) and the *35 U.S.C. 103(a)* rejection of claims 5-6 and 11-13 as being unpatentable over U.S. Patent No. 5,681,645 to Strack are noted. The two references are actually the same because the '645 patent issued on the priority application of the PCT application. Note column 8, lines 58-67, column 6, lines 22-67, column 9, lines 14-23, column 9, lines 35-61, and column 11, lines 42-46 of the '645 patent, all are cited by the Examiner, which exactly correspond to page 8, lines 7-13, page 8 line 29 through page 9 line 28, page 12 line 30 through page 13 line 2, page 13, lines 10-29, and page 16, lines 8-12 of WO 96/38620, respectively. The Examiner factually applied a single reference (hereinafter Starck) in both rejections. Applicants will proceed accordingly.

As to claims 1-4, 7-10 and 14, the Examiner rejects these claims under *35 U.S.C. 102(b)* because Strack describes the *essential* limitations of the claimed invention. It does not matter how many essential limitations of the claimed invention a reference teaches. "A claim is anticipated only if *each and every* element as set forth in the claim is found, either expressly or inherently described, in a single prior art reference." *Verdegaal Bros. v. Union Oil Co. of California*, 814 F.2d 628, 631, 2 USPQ2d 1051, 1053 (Fed. Cir. 1987) (emphasis added). Strack, *as admitted by the Examiner* in page 5, lines 8-9 of the Office Action, fails to teach or disclose the limitation of

orthogonal stretchability recited in independent claim 1. The *35 U.S.C. 102(b)* rejection of claims 1-4, 7-10 and 14 is not well founded and should be withdrawn or rephrased.

As to claims 5-6 and 11-13, the Examiner discusses at length how Strack provides numerous teachings without specifying how the teachings are readable on or at least related to the claim limitations. This makes it very difficult to understand the Examiner's rejection. However, the following supposed errors are noted in the Examiner's rationale.

As to claims 5-6, the Strack teachings cited by the Examiner fail to disclose, teach or suggest the claimed component fibers of the *fibrous assembly* of the present invention. For example, column 5, lines 58-67 of the '645 patent discloses a *non-woven elastomeric web* which at best corresponds to the elastic of the present invention rather than to the non-elastic fibrous assembly claimed in claims 5-6.

As to claim 11, the Examiner's positions as taken in paragraphs 16 and 19 of the Office Action appear contradictory. In paragraph 16, the Examiner states that it is not clear what is encompassed by JIS-L-1092 and JIS-Z-0208, i.e., the scope of claim 11 is unascertainable. However, in paragraph 17, the Examiner seems to hold that the limitations of unascertainable claims 11 are nevertheless found in Strack. Applicants respectfully submit that the reference fails to disclose, teach or suggest the limitations presently claimed in claim 11.

The *35 U.S.C. 103(a)* rejection of claims 5-6 and 11-13 is therefore erroneous and should be withdrawn.

Claims 15-19 have been added to recite other features of the present invention not found in nor suggested by the applied art of record.

The applied reference teaches a laminate material made from an elastomeric web and a web of textile material discontinuously adhesively bonded to each side of the elastomeric web. The

textile web may be knits, wovens or scrim materials. *See* Abstract of Strack. Thus, fibers of the textile web must be somehow connected to each other, i.e., by interlacing or interlocking in an identifiable manner, even in regions where the textile web is not bonded to the elastomeric web. In other words, the fibers of the textile web are not separate from each other and will prevent each other from being free to reorientate and extend when the composite sheet is stretched. As a result, the stretchability of the composite sheet is substantially affected by the stretchability of the textile fabric, and the stretchability of the composite sheet cannot be obtained as high as in the claimed device.

In contrast, the fibrous assembly of the present invention contains component fibers which are separate one from another, as described in page 5, lines 5-6 and page 11, lines 4-7 from bottom, of the specification. Consequently, the inelastic layer has little influence, if at all, on the stretchability of the composite sheet so as the stretchability of the composite sheet may be achieved as high as possible. *See* page 2, lines 12-15 from bottom, page 5 lines 5-6, page 9 lines 2-5 of the specification. This feature has been included in the new claims to further define the claimed invention over the applied reference.

More particular, new independent claim 15 is directed to a composite sheet comprising an elastic sheet intermittently bonded to a *non-woven* inelastic fibrous assembly. Claim 15 finds solid support in the original specification, especially pages 11-18 where methods of producing the composite sheet are disclosed. Strack fails to disclose, teach or suggest this feature because one of the Strack webs is a *textile* web.

Claim 16 requires that the component fibers overlay each other *without being interlaced or interlocked* with each other. Claim 16 finds solid support in the original specification, especially page 11 lines 13-20, and page 13, line 4 from bottom through page 14, line 2. Strack fails to disclose, teach or suggest this feature because the fibers in the Strack textile web are interlaced or interlocked.

Claims 17-19 are patentable over the applied reference as will be apparent to the Examiner upon reviewing the new claims.

Each of the Examiner's rejections has been traversed. Accordingly, Applicants respectfully submit that all claims are now in condition for allowance. Early and favorable indication of allowance is courteously solicited.

The Examiner is invited to telephone the undersigned, Applicant's attorney of record, to facilitate advancement of the present application.

Respectfully submitted,
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MARKED-UP VERSION SHOWING CHANGES

Fig. 3 is a view similar to Fig. 1 but showing another embodiment of this invention;

Fig. 4 is a diagram schematically illustrating an example of the process for making the composite sheet; ~~and~~

Fig. 5 is a diagram similar to Fig. 4 but illustrating another example of the process for making the composite sheet;

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Details of an elastically stretchable composite sheet according to this invention will be more fully understood from the description given hereunder with reference to the accompanying drawings.

An elastically stretchable composite sheet 1 shown by Fig. 1 in a perspective view comprises an upper layer 2 and a lower layer 3 bonded together at their bond regions 4. The composite sheet 1 is elastically stretchable and contractable at least in a direction represented by a double-headed arrow Y - Y rather than in a direction indicated by a double-headed arrow X - X being orthogonal to the direction Y - Y, as indicated by imaginary lines.

The upper layer 2 of the composite sheet 1 is inelastically stretchable at least in the direction Y - Y rather than in the direction X - X. This upper layer 2 is an assembly

Fig.6 is a schematic view of a testing device used in a testing method in accordance with Japanese Industrial Standards JIS L 1092; and Fig.7 is ——————— JIS Z 0208.

of long, preferably, continuous thermoplastic synthetic resin fibers 6 which continuously extend in a region defined around the bond regions 4. The fibers 6 are sealed with one another only at the bond regions 4 and neither sealed nor bonded together, i.e., separated one from another in the region defined around the bond regions 4. Between each pair of the adjacent bond regions, the continuous fibers 6 extend over the upper surface of the lower layer 3 along irregular curves. As the composite sheet 1 is stretched in the direction Y - Y and/or in the direction X - X, the curved continuous fibers 6 are reoriented to extend in the direction Y - Y and/or in the direction X - X and the upper layer 2 is inelastically stretched. Such continuous fibers 6 may be of inelastic synthetic resin such as polypropyrene, ethylene-propyrene random copolymer, ethylene-propyrene-butene random copolymer, polyester or polyethylene. Each of the continuous fibers 6 preferably have a diameter of 0.1 - 50 μm.

The lower layer 3 of the composite sheet 1 comprises a sheet which is elastically stretchable in the direction Y - Y, preferably both in the direction Y - Y and in the direction X - X. This sheet has a stretch ratio of at least 200 %, preferably at least 400 % in the direction Y - Y and elastically contractile again to less than 1.3 times of its initial length after

stretched by 100 %. Such sheet may be a fibrous assembly comprising continuous elastic fibers made of thermoplastic elastomer, a nonwoven fabric comprising the continuous fibers mechanically entangled or bonded with together or a film made of thermoplastic elastomer. The film, if it is used, may be moisture-permeable or moisture-impermeable. The sheet is exemplarily shown in the form of nonwoven fabric comprising continuous fibers 10.

The continuous fibers or film constituting the lower layer 3 may be made of block copolymerized polyester comprising hard and soft ingredients. The continuous fibers made of such polyester is able to cope with an environmental condition with its high moisture absorption as well as its high moisture permeability comparable to those of natural fibers. Therefore, the composite sheet 1 comprising such continuous fibers is suitable as stock material for garments such as disposable gowns. The film made of such polyester is also adequately moisture-permeable and the composite sheet 1 using the film is suitable as stock material for liquid-impervious backsheets of disposable body fluid absorbent articles such as disposable diapers or sanitary napkins. For such application of the film, its hard and soft ingredients are combined so that the film may have a moisture permeability of at least 1000 g/m^2/24hrs. as

measured in accordance with the prescription of JIS (Japanese Industrial Standards) Z 0208 and a water pressure resistance of at least 1 m, preferably at least 2 m as measured in accordance with the prescription of JIS L 1092. An example of the film useful to make the composite sheet 1 is a product of Toyobo Co., Ltd. put on the market under a trade name of PELPRENE P30B.

The hard ingredient is preferably polyester presenting a glass transition temperature of 50℃ or higher which is obtained from dicarboxylic acid and diol. At least one of dicarboxylic acid and diol preferably includes an aromatic ring. The dicarboxylic acid includes aromatic dicarboxylic acid such as phthalic acid, terephthalic acid, isophthalic acid, 2, 6-naphtalene dicarboxylic acid, 1, 5-naphtalene dicarboxylic acid, diphenyl-4, 4'-naphtalene dicarboxylic acid, 3, 3'-dimethyldiphenyl-4, 4'-dicarboxylic acid, 4, 4'-diphenylsulfonedicarboxylic acid, and 4, 4'-diphenoxyethanedicarboxylic acid; aliphatic dicarboxylic acid such as succinic acid, oxalic acid, adipic acid, sebacic acid and dodecane diacid; and alicyclic dicarboxylic acid such as 1, 4-cyclohexanedicarboxylic acid and decahydronaphthalene-2, 6-dicarboxylic acid.

The soft ingredient is preferably polyether, aliphatic polyester or copolymer of polyether and aliphatic polyester,

The testing methods used in JIS Z 0208 and JIS L 1092 will be described ~~after~~ below with reference to Figs. 6 and 7

the area of the individual bond regions 4 may be selectively varied in a range of 0.03 ~ 10 mm^2 and/or the total area of these bond regions 4 may be selectively varied in a range of 1 - 50 % of the area of the second composite web 44 without departing from the scope of this invention.

The steps illustrated by Fig. 5 can be modified so that the third melt blown fiber molder is used to feed the third continuous fibers onto the lower surface of the film 52 onto which the first continuous fibers 35 have been fed to obtain the composite sheet 1 of Fig. 3 having the film 52 sandwiched between two fibrous webs.

In the following section, a testing method in accordance with JIS L 1092 will be described with reference to Fig. 6.

Insert JIS-L-1092 here

In the following section, a testing method in accordance with JIS Z 0208 will be described with reference to Fig. 7.

Insert JIS-Z-0208 here

WHAT IS CLAIMED IS:

1. An elastically stretchable composite sheet, comprising an elastic sheet having a stretchability in two directions orthogonal to each other; and a sheet-like fibrous assembly having an extensibility in said two directions bonded to at least one surface of said elastic sheet, wherein:

said fibrous assembly has a inelastic extensibility, said elastic sheet and said fibrous assembly are bonded together at bond regions arranged intermittently in said two directions, and component fibers ~~constituting~~ said fibrous assembly are long fibers continuously extending and describing curves between ~~each pair of~~ adjacent bond regions in which said long fiber is bonded to said elastic sheet.

2. The composite sheet according to Claim 1, wherein said component fibers are neither sealed nor bonded with one another in a region ~~extending~~ between ~~each pair of~~ adjacent said bond regions.

3. The composite sheet according to Claim 1, wherein said component fibers are independent one from another in said regions ~~extending between each pair of adjacent said bond regions~~.

4. The composite sheet according to Claim 1, wherein said component fibers describe loops in said region~~s extending between each pair of adjacent said bond regions~~. bond-free

5. The composite sheet according to Claim 1, wherein said component fibers are ~~in the form of~~ stretched yarns made of polypropyrene or polyester.

6. The composite sheet according to Claim 1, wherein each of said component fibers has a diameter of 0.1 ~ 50μm.

7. The composite sheet according to Claim 1, wherein said component fibers are continuous fibers.

8. The composite sheet according to Claim 1, wherein said fibrous assembly has a basis weight of 2 ~ 100 g/m^2.

9. The composite sheet according to Claim 1, wherein said elastic sheet is made of an elastically stretchable film or elastically stretchable continuous fibers.

10. The composite sheet according to Claim 9, wherein said

elastically stretchable film is moisture-permeable.

11. The composite sheet according to Claim 9, wherein said elastically stretchable film presents a moisture-permeability of at least 1000 g/m²/24 hrs as measured according to ~~the prescription of~~ JIS Z 0208, and a water pressure resistance of at least 1 m as measured according to ~~the prescription of~~ JIS L 1092.

12. The composite sheet according to Claim 9, wherein said elastically stretchable film is made of block copolymerized polyester comprising hard and soft ingredients and said soft ingredient is polyether or copolymer of polyether.

13. The composite sheet according to Claim 9, wherein said elastically stretchable continuous fibers are made of block copolymerized polyester comprising hard and soft ingredients and said soft ingredient is polyether or copolymer of polyether.

14. The composite sheet according to Claim 1, wherein said composite sheet is elastically stretchable at least by 20 % in said two directions.

EXHIBIT A

#6/A Cont

JIS

JAPANESE INDUSTRIAL STANDARD

Translated and Published by
Japanese Standards Association

40 pages total

JIS L 1092 : 1998

Testing methods for water resistance of textiles

ICS 59.080.01

Descriptors : textile products, cloth, water-resistance tests, waterproof materials

Reference number : JIS L 1092 : 1998 (E)

L 1092:1998

Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently **JIS L 1092 : 1992** is replaced with **JIS L 1092 : 1998**.

Date of Establishment: 1977-03-01

Date of Revision: 1998-06-20

Date of Public Notice in Official Gazette: 1998-06-22

Investigated by: Japanese Industrial Standards Committee

Divisional Council on Consumer Life

JIS L 1092:1998, First English edition published in 1999-04

Translated and published by: Japanese Standards Association
4-1-24, Akasaka, Minato-ku, Tokyo, 107-8440 JAPAN

In the event of any doubts arising as to the contents, the original JIS is to be the final authority.

Printed in Japan

Testing methods for water resistance of textiles

Introduction This Japanese Industrial Standard was made on the basis of **ISO 811**, *Textile fabrics—Determination of resistance to water penetration—Hydrostatic pressure test* published in 1981 as the first edition, **ISO 4920**, *Textiles—Determination of resistance to surface wetting (spray test) of fabrics* published in 1981 as the first edition and **ISO 9865**, *Textiles—Determination of water repellency of fabrics by the Bundesmann rain-shower test* published in 1991 as the first edition without changing the technical contents with the exception of the following items added.

a) Definitions, sampling of test specimens and conditioning specified conventionally in the Japanese Industrial Standard were added.

b) B method of the water penetration test (hydrostatic pressure method, constant hydraulic pressure method and water leakage method) specified conventionally in the Japanese Industrial Standard was added.

c) A method of the water penetration test (constant hydraulic pressure method and water leakage method) and B method of the rain test specified conventionally in the Japanese Industrial Standard were also added.

1 Scope This Japanese Industrial Standard specifies the testing methods for water resistance of textiles.

Remarks 1 Water resistance is a generic term of water proofing, resistance to surface wetting, water leakage, etc.

2 The following standards are the corresponding International Standards to this Standard:

ISO 811: 1981	*Textile fabrics—Determination of resistance to water penetration—Hydrostatic pressure test.*
ISO 4920: 1981	*Textiles—Determination of resistance to surface wetting (spray test) of fabrics*
ISO 9865: 1991	*Textiles—Determination of water repellency of fabrics by the Bundesmann rain-shower test*

2 Normative references The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The latest editions of them apply.

JIS K 0117	*General rules for infrared spectrophotometric analysis.*
JIS K 1521	*Perchloroethylene*
JIS K 2246	*Rust preventive oils*
JIS K 8322	*Chloroform*
JIS K 8848	*Hexane*
JIS K 8858	*Benzene*
JIS K 8891	*Methanol*

JIS L 0105 *General principles of physical testing methods for textiles*
JIS L 0217 *Care labelling of textile goods*
JIS L 0803 *Standard adjacent fabrics for staining of colour fastness test*
JIS L 1096 *Testing methods for woven fabrics*
JIS P 3801 *Filter paper (for chemical analysis)*
JIS R 3503 *Glass apparatus for chemical analysis*
JIS Z 8806 *Humidity—Measurement methods*

3 Definitions For the purpose of this Standard the following definitions apply:

a) **standard condition of laboratory** The condition that the temperature, humidity and the tolerance on them in laboratory are the temperature of (20±2) ℃ and the relative humidity of (65±2) % which are specified in **JIS L 0105**.

Remarks: Use a Meteorological Agency type or Assmann's aspiration psychrometer as specified in **JIS Z 8806** to determine the temperature and use the humidity table by Sprung's formula to determine the relative humidity.

b) **standard condition of test specimen** The condition under which the test specimens are allowed to stand and come to a constant weight.

c) **constant weight** The mass of the test specimen when weighed at intervals not shorter than one hour and thus obtained difference between two measurements has come to within 0.1 % of the mass at the latter measurement.

4 Classification of tests The tests shall be classified as shown below:

a) **Test for water penetration (Hydrostatic pressure method)** This test applies mainly to textile fabrics with no air permeability.

1) Method A (Low hydraulic pressure method)
2) Method B (High hydraulic pressure method) (1)

Note (1) This method usually applies to the test specimens which can be tested by applying a hydraulic pressure exceeding 10 kPa.

b) **Test for resistance to surface wetting (Spray method)** This test applies to textile fabrics with air permeability.

c) **Rain test (Shower test) method A**

5 Sampling and conditioning of test specimens

5.1 Sampling and preparation of test specimens The test specimens shall be sampled at random from the area distant $\frac{1}{10}$ of width from both selvages and not less than 1 m from the end in the case of woven fabric or kilted fabric while, in the case of product, be sampled from the front cloth of the fabric, and they shall be brought to standard condition.

Remarks: If it is impossible to keep the laboratory at standard condition, place the test specimen in a closed container (containing 36 % sulfuric acid) maintained at (20±2) ℃ and bring to a constant weight.

5.2 Conditioning of test specimens If necessary, carry out the following conditionings on the test specimens specified in 5.1 separately or in combination and then carry out the tests specified in 6.

Remarks: The waterproof retention of the conditioned test specimens shall be calculated by the following formula down to one decimal place after testing the test specimen before conditioning and after conditioning in accordance with the methods specified in 6.

$$\text{Waterproof retention (\%)} = \frac{A}{A_0} \times 100$$

where, A_0: test value of non-conditioned test specimen

A: test value of conditioned test specimen

a) **Laundry treatment** One of the following methods is used:

1) **Method A** (method to use a stirring type washing machine) Use the method specified in 6.23.1 of **JIS L 1096** and use drip dry as drying method.

2) **Method B** (method to use a cylinder type washing machine) Use the method specified in 6.23.2 of **JIS L 1096**.

3) **Method C** (method to use an electric washing machine for domestic use) Use the method specified in No. 103 of Attached Table 1 of **JIS L 0217**.

b) **Dry cleaning treatment** Put approximately 3.78 *l* of perchloroethylene (²) at approximately 30 ℃ in the cylinder of wash cylinder type washer as shown in Fig. 1, insert an approximately 50 cm × 50 cm test specimen and a load cloth (³) adjusted to become approximately 0.45 kg in their combined weight and operate the washer for 10 minutes.

Dehydration is carried out by a centrifuge until almost all water is removed. If it is impossible to do so, press lightly to remove water, place between filter papers or cloth and press again to remove the water. Do not use a mangle to squeeze.

Dry using any one selected from among the following four methods:

Notes (²) The perchloroethylene is as specified in **JIS K 1521**.

(³) The cotton cloth (standard adjacent fabric for staining No. 3) specified in **JIS L 0803** whose edges are hemmed and size is the same as the test specimen is used.

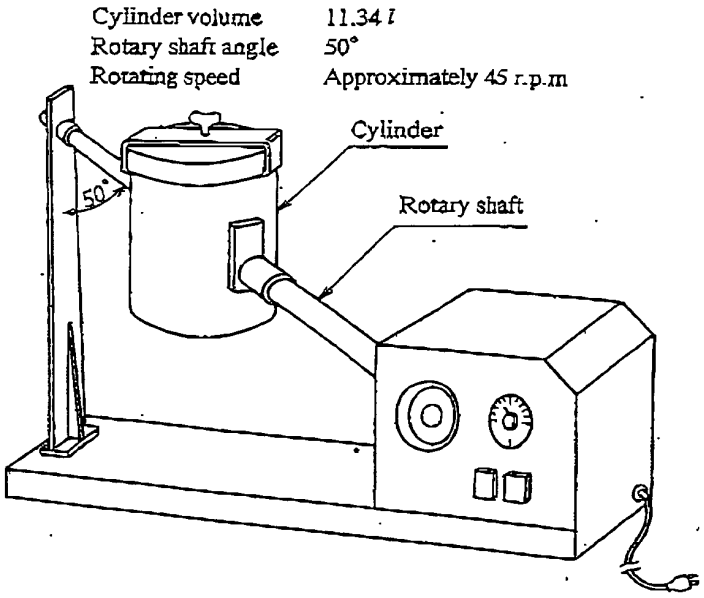


Fig. 1 Wash cylinder type washer

1) **Screen drying** After centrifuging, dry the test specimen by smoothing unnatural wrinkles without twisting or elongating, and spreading on a flat screen mesh or a surface drilled with similar holes. Natural drying shall be used. In order to enhance the efficiency of the test, it may be dried in a dryer at not higher than 70 ℃.

2) **Line drying** After centrifuging, grip two corners so that the warp or the wale direction is vertical and dry by suspending in a place without ventilation at room temperature.

Remarks: Line drying should not be used for fabrics likely to be elongated easily in the wale direction.

3) **Drip drying** Without centrifuging the test specimen, grip two corners so that the warp or the wale direction is vertical and dry by suspending in a place without ventilation at room temperature.

Remarks: Drip drying is used for wash and wear fabrics.

4) **Tumble drying** After centrifuging, put in a tumble dryer and operate at the temperature of 50 ℃ to 70 ℃ for 30 minutes or until the test specimen dries.

c) **Weathering treatment** Perform the method specified in **5.36** of **JIS K 2246**. The time taken for one time of treatment shall be 20 hours.

6 Testing methods

6.1 Test for water penetration (Hydrostatic pressure method) The test shall be carried out by the hydrostatic pressure method in accordance with the following method A (Low

hydraulic pressure) or method B (High hydraulic pressure method).

In addition to the hydrostatic pressure method, the constant hydraulic pressure method or water leakage method specified in Annex 1 may be used.

a) Method A (Low hydraulic pressure method) is as follows:

1) **Apparatus and material** The following apparatus and material shall be used:

1.1) **Water penetration test apparatus (for low hydraulic pressure)** The apparatus shown in Fig. 2 or those equivalent thereto whose rate of increase of water pressure is (60±3) cm/min and (10±0.5) cm/min. The clamp shall be of such size that the part of the test specimen which touches the water is 100 cm^2.

1.2) **Hydraulic pressure gauge (manometer)** With 0.5 cm scale and of approximately 1 m or higher in the maximum water level when raising the water levelling apparatus.

1.3) **Water** The distilled or ion-exchange water maintained at (20±2) ℃ in the test shall be used and the chosen alternative shall be stated in the test report.

Informative reference: Water temperature influences sometimes the test result.

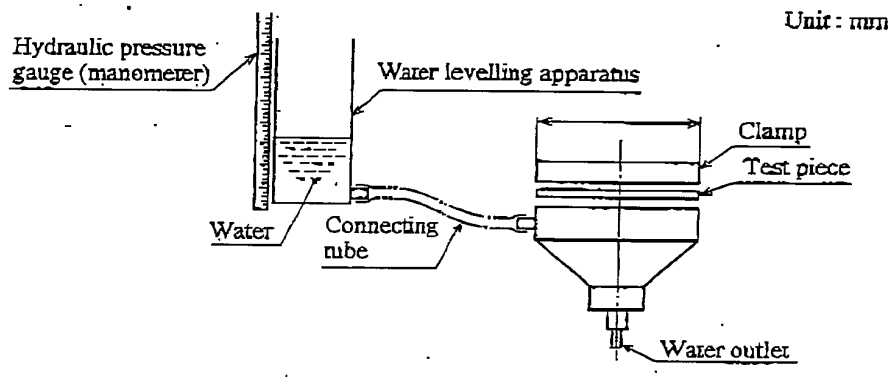


Fig. 2 Water penetration test apparatus (for low hydraulic pressure)

2) **Procedure** From the test specimens specified in 5, sample test pieces each measuring approximately 15 cm × 15 cm, take five sheets of them for the following each test, mount the test pieces on the water penetration test apparatus shown in Fig. 2 in such a way that the front surface (*) touches the water, raise the water level by raising the water levelling apparatus containing water at a speed of (60±3) cm/min or (10±0.5) cm/min and measure the water level to an accuracy of cm at the time when the water comes out from three places on the reverse surface of the test pieces. The accuracy of the water level to be reported is

- less than 1 m: 0.5 cm
- from 1 m and less than 2 m: 1.0 cm
- 2 m or more: 2.0 cm

Each result of five test pieces and the average value shall be expressed down to first decimal place. If the water does not come out from three places even when the water

level is raised, measure the water level at the time when the water comes out from one or two places and state that effect in the test report.

The extremely small water drops which do not grow after being formed and the waterdrops formed by penetrating through the same place shall be ignored.

Note (*) The front surface is the waterproofed surface or the surface the water touches at the time of use.

Informative reference: The test for water penetration, method A (Low hydraulic pressure method) is the same test method as that in **ISO 811**.

b) Method B (High hydraulic pressure method) is as follows:

1) **Apparatus and material** The following apparatus and material shall be used:

1.1) **Water penetration test apparatus (for high hydraulic pressure)** The apparatus shown in Fig. 3 or those equivalent thereto whose rate of increase of water pressure is 100 kPa per minute.

1.2) **Water** The distilled or ion-exchange water maintained at (20±2) ℃ in the test shall be used and the chosen alternative shall be stated in the test report.

2) **Procedure** From the test specimens specified in **5**, sample the test pieces each measuring approximately 15 cm × 15 cm, take five sheets of them for the following each test, mount the test pieces on the water penetration test apparatus shown in Fig. 3 in such a way that the front surface (*) touches the water, fill the cylinder with water, turn the piston handle to apply hydraulic pressure at a rate of 100 kPa per minute and measure the water pressure (kPa) to an accuracy of $\frac{1}{2}$ of the scale of the pressure indicator at the time when the water comes out from three places on the reverse surface of the test pieces.

The average value of five measurements shall be expressed down to first decimal place. If the water does not come out from three places even when the hydraulic pressure is raised, measure the water level at the time when the water comes out from one or two places and state that effect in the test report.

The extremely small water drops which do not grow after being formed and the waterdrops formed by penetrating through the same place shall be ignored.

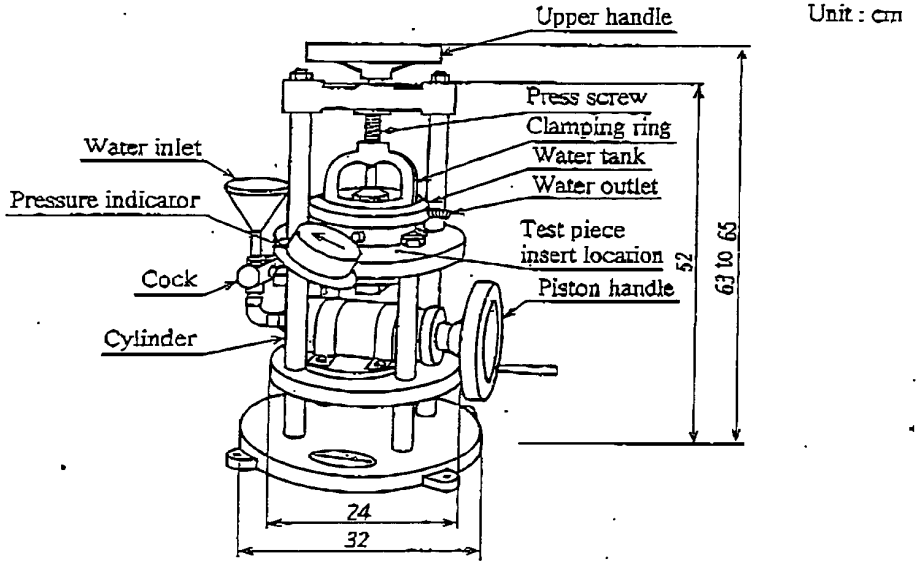


Fig. 3 Water penetration test apparatus (for high hydraulic pressure)

6.2 Test for resistance to surface wetting (Spray test) The test for resistance to surface wetting shall be as follows :

a) **Apparatus and material** The following apparatus and material shall be used :

1) **Apparatus for test for resistance to surface wetting** The apparatus shown in Fig. 4 or those equivalent thereto with the glass funnel of 250 ml or larger in volume and the spray nozzle capable of spraying 250 ml of water in 20 seconds to 30 seconds.

2) **Test piece holder** The metal holder of 150 mm diameter or that equivalent thereto.

3) **Comparison examples for wetting condition** The examples to which the rating number is given according to the wetting conditions as shown in Fig. 5.

4) **Water** The distilled or ion-exchange water maintained at (20±2) ℃ in the test shall be used and the chosen alternative shall be stated in the test report.

b) **Procedure** From the test specimens specified in 5, sample three sheets of test pieces measuring approximately 20 cm × 20 cm, mount a test piece on the holder without creasing, make the center of spray coincide with the center of the test piece holder using the apparatus for the test of resistance (see Fig. 4) to surface wetting so that the warp direction of the test piece is parallel to the flow of water down. Pour 250 ml of water into the glass funnel to spray on the test piece within 25 seconds to 30 seconds.

Remove the holder from the stand, hold horizontally with its one end, face the front surface of the test piece downward, tap another end against a solid object, turn by 180° and repeat the same procedure as before to drop the excessive water drops. Compare the wetting condition of the test piece as attached to the holder with the comparison example for wetting condition specified in Fig. 5 to assess. No middle rating is taken.

Informative reference: The test for the resistance to surface wetting (Spray test) is the same test method as specified in **ISO 4920**.

Unit : mm

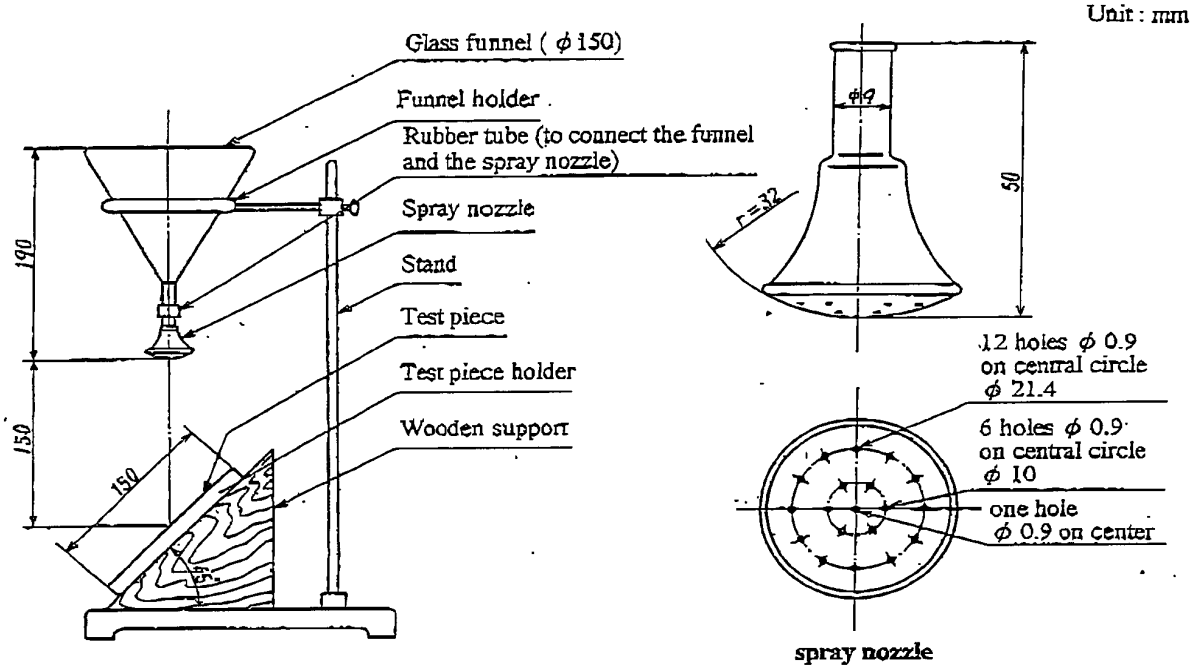
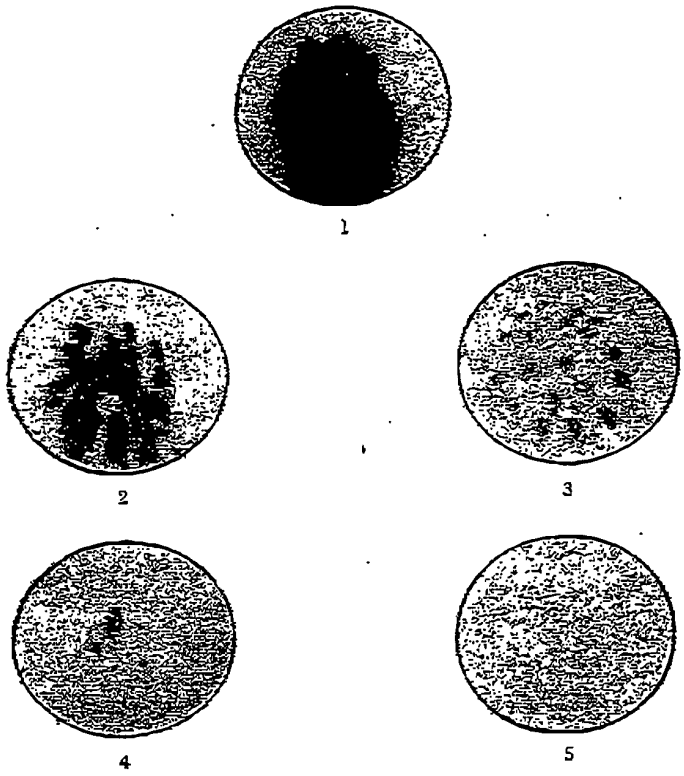


Fig. 4 Apparatus for the test of resistance to surface wetting



1: Complete wetting of whole upper surface.

2: Half wetting of whole of upper surface and small individual wetting of penetrating the cloth.

3: Wetting of upper surface at small spray points.

4: Slight random sticking or wetting of upper surface.

5: No sticking or wetting of upper surface.

Fig. 5 Comparison sample of wetting condition

6.3 Rain test (Shower test) method A The rain test method A shall be as follows: In addition to the method A, the method B specified in Annex 1 may be used.

a) **Apparatus and material** The apparatus and material shall be as follows:

1) **Bundesman rain-shower test apparatus** The apparatus shown in Fig. 6 or those equivalent thereto which consist of rain shower equipment and testing device. The rain shower equipment comprising a system of about 300 identical drop-forming nozzles (nozzle of 4 mm diameter to produce a waterdrop of approximately 0.07 ml) over a circular surface (area of approximately 1 300 cm^2) of 406 mm diameter shall be capable of producing a rain shower of the flow rate of (100±5) ml/min per 100 cm^2.

The testing device shall be so constructed that four cups are mounted on the stand for the centerline of the cup to be inclined at 15° from the vertical and the stand rotates

approximately six turns in one minute. Each test cup, the outer diameter of which is 100 mm, enables the specimen (tested area of 80 cm^2) to be mounted on the upper part by means of a test piece clamping ring and the water passing through the test piece to be collected in the cup. In addition, each cup comprises a polished stainless steel wiper of 48 mm in length (Informative reference: Curved slightly to the top in the longitudinal direction with radius of curvature 630 mm), 5 mm in width and 5 mm in radius of rounded scraper edge. The wiper is pressed against the underside of the test piece during testing with force of 2.5 N to describe 20 reciprocating rotary movements per minute at an angle of 100°. The distance between the waterdrop-forming nozzle of rain shower equipment and the center of the test piece mounted on the testing device shall be 1 500 mm.

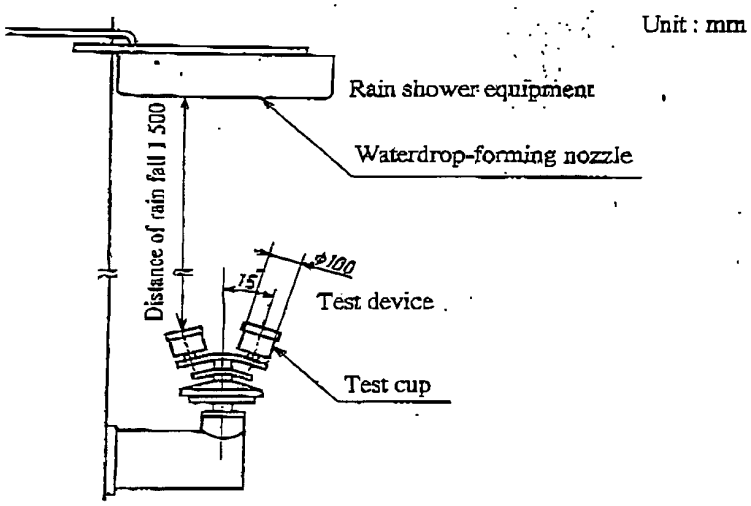
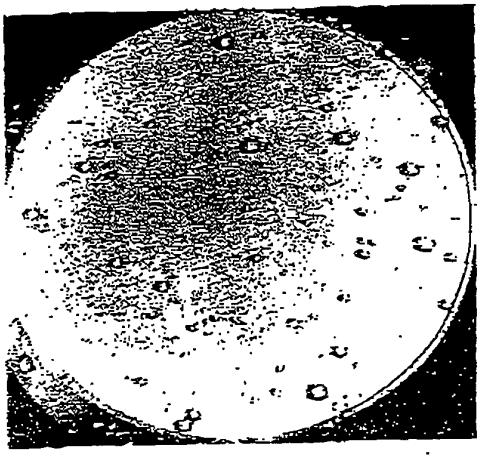


Fig. 6 Bundesmann rain-shower test apparatus

2) **Comparison samples for wetting condition** The reference samples specifying the assessment according to the wetting conditions as illustrated in Fig. 7.



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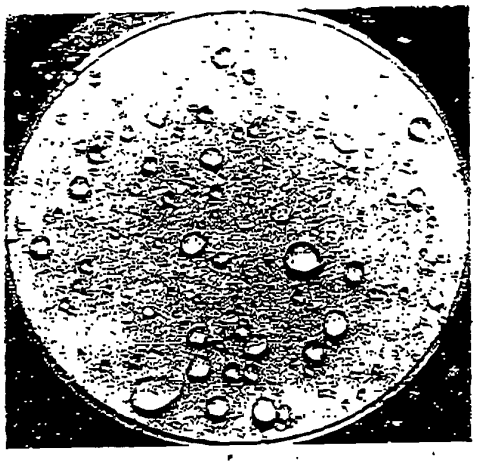

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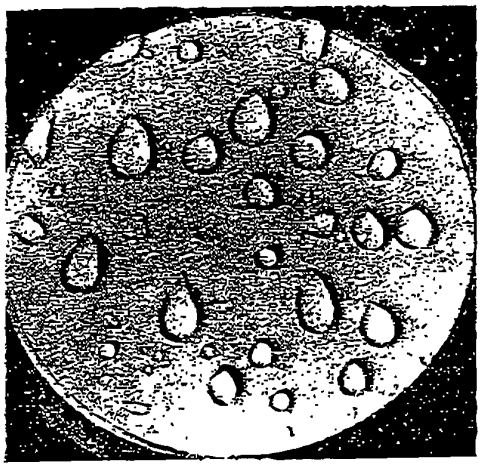

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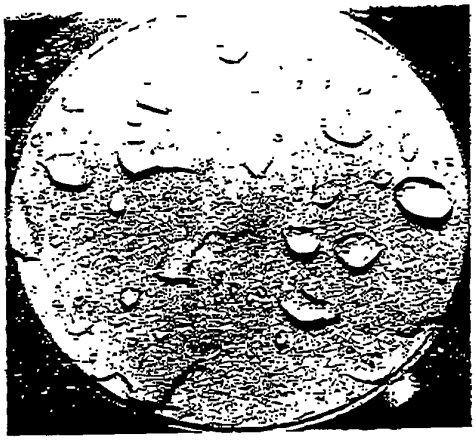
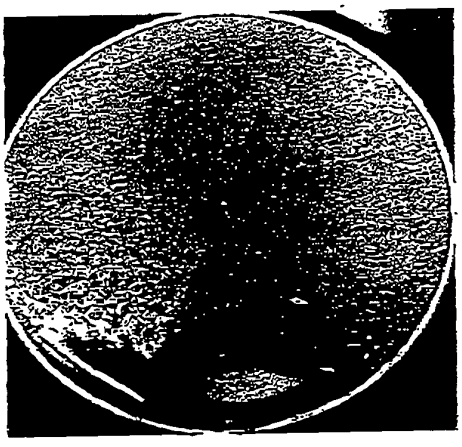


Grade 2



Grade 1

Fig. 7 Refernce photographs for comparison of wetting condition

3) **Centrifuge** Comprising a disc with a horizontal test piece mounting surface of diameter 175 mm rotating at 700 rpm (the time required to attain this speed is between 1 s and 2 s). The total mass of the rotating part shall be 410 g. On the test piece mounting surface, approximately 50 ribs (1 mm in height) are provided in a radial arrangement, and four steel pins of 6 mm long spaced 60 mm away from the center of the rotary axis are provided in an equally spaced arrangement to secure the test pieces.

4) **Balance** Capable of weighing to an accuracy of 0.01 g.

5) **Water** Temperature of (20±3) ℃. Temperature, hardness and pH during the test shall be recorded.

b) **Preparation of rain-shower** After checking each part of the test apparatus, switch on the rain shower equipment for approximately 15 minutes to set the rain flow control valve of the rain shower equipment so that (200 ±10) ml of water is collected in each cup after 2.5 minutes.

c) **Procedure** From the specimens specified in 5, sample four sheets of circular test pieces of 140 mm diameter, weigh their mass to an accuracy of 0.01 g, mount on the test cups and start the operation after fixing them on the stand of the test device. After exposing the test pieces to the rain-shower for 10 minutes (rain falling time may be 1 min or 5 min), stop rain falling. Assess the wetting condition of upper surface of the test pieces by visual comparison with the reference samples, remove the test pieces from the cups, mount on the centrifuge, operate for 15 seconds to remove the excessive drops on the test pieces and weigh immediately the mass to an accuracy of 0.01 g. Then measure the volume of the water which has passed through the test pieces and is collected in the cups to determine the leakage (ml).

The test results shall be expressed, by calculating the amount of water absorption (g) and the rate of water absorption (%) by the following formulas, with the average of four sheets to the integer place for the water repellency, down to two decimal places for the amount of water absorption and down to one decimal place for the rate of water absorption.

$$\text{Amount of water absorption (g)} = M - M_0$$

$$\text{Rate of water absorption (\%)} = \frac{M - M_0}{M_0} \times 100$$

where M_0: mass of test pieces before the test (g)

M: mass of test pieces after the test (g)

Informative reference: The rain test (Shower test) is the same test method as that in ISO 9865

7 **Test report** The test report shall include the following information. If the conditioning of specimen specified in 5.2 is carried out, the method of conditioning, number of times of conditioning and circumstances at the time of conditioning shall be stated as well.

a) **Test for water penetration (Hydrostatic pressure method)**

1) Classification of test

2) Measured values: water level (method A) or water pressure (method B) when water comes out from three places

b) **Test for resistance to surface wetting (Spray test)** Result of assessment of each test specimen

c) **Rain test (Shower test) method A**

1) Amount of water absorption and rate of water absorption

2) Wetting condition of surface

3) Water leakage

4) Temperature, hardness and pH of the water used for the shower

5) Amount of rain fall and time of rain falling, where appropriate

Annex 1 (normative) Testing methods for water resistance

1 Scope This Annex specifies the test for water penetration based on constant hydraulic pressure method or water leakage method and the rain test method B which have been specified conventionally in the Japanese Industrial Standard.

2 Classification of tests The tests shall be classified as given in the following:

a) **Test for water penetration (Constant hydraulic pressure method or Water leakage method)** This test applies mainly to textile fabrics with no air permeability.

1) Method A (Low hydraulic pressure method)

2) Method B (High hydraulic pressure method) (1)

Note (1) Usually, this test applies to the specimens capable of being tested applying hydraulic pressure of 10 kPa or higher.

b) **Rain test (Shower test) method B**

3 Test methods

3.1 Test for water penetration (Constant hydraulic pressure method or Water leakage method) The test shall be carried out by the constant hydraulic pressure method or the water leakage method in accordance with Method A (Low hydraulic pressure method) or Method B (High hydraulic pressure method).

a) Method A (Low hydraulic pressure method) shall be as follows:

1) **Apparatus and material** The following apparatus and material shall be used:

1.1) **Apparatus for water penetration test (for low hydraulic pressure)** The same apparatus as in **6.1 a) 1.1)** in the body.

1.2) **Hydraulic pressure gauge (manometer)** The same gauge as in **6.1 a) 1.2)** of the body.

1.3) **Stopwatch** With a 0.5 s scale.

1.4) **Measuring cylinder** With a 1 ml scale.

1.5) **Water** Distilled or ion-exchange water maintained at (20±2) ℃ in the test shall be used and the chosen alternative shall be stated in the test report.

2) **Procedure** From the test specimens specified in **5**, sample the test pieces each measuring approximately 15 cm×15 cm, take five sheets of them for the following each test, mount the test pieces on the apparatus for water penetration test (for low hydraulic pressure) in such a way that the front surface (2) touches the water, raise the water levelling device filled with water at a rate of (60±3) cm/min or (10±0.5)

cm/min, obtain the resistance to water penetration by either of the following methods and express the average value of five measurements down to one decimal place. In this case, the method used shall be stated.

Note (2) Front surface is the waterproofed surface or the surface the water touches at the time of the use.

2.1) **Constant hydraulic pressure method** When the water is raised to a given level and allowed to stand as it is, the time required until the water comes out from the reverse side of the test piece at three places is measured to an accuracy of 0.5 s. In the test result, the given water level shall be stated. If the water does not come out at three places, the time required until the water comes out at one place or two places is measured or the measurement is made by changing the water level higher. The change of water level shall be also stated in the report.

The extremely small waterdrops which do not grow after being formed shall be ignored.

2.2) **Water leakage method** After raising the water level up to a given level, the volume (ml) of the water passing through the test piece is measured after the given time has passed by putting in a measuring cylinder. The volume per unit area (cm^2) is expressed. In the test result, the given water level and the given time shall be stated.

b) Method B (High hydraulic pressure method) shall be as follows:

1) **Apparatus and material** The following apparatus and material shall be used.

1.1) **Apparatus for water penetration test (for high hydraulic pressure)** The equivalent of the apparatus in **6.1 b) 1.1)** of the body.

1.2) **Stopwatch** With a 0.5 s scale.

1.3) **Measuring cylinder** With a 1 ml scale.

1.4) **Water** Distilled or ion-exchange water maintained at (20±2) ℃ in the test shall be used and the chosen alternative shall be stated in the test report.

2) **Procedure** From the test specimens specified in 5, sample the test pieces each measuring approximately 15 cm × 15 cm, take five sheets of them for the following each test, mount them on the apparatus for water penetration test (for high hydraulic pressure) in such a way that the front surface (1) touches the water, fill the cylinder with water, turn the piston handle to apply hydraulic pressure at a rate of 100 kPa per minute, obtain the resistance to water penetration by the following each method and express the average value of five measurements down to one decimal place. In this case, the method used shall be stated.

2.1) **Constant hydraulic pressure method** When a given hydraulic pressure is applied and allowed to stand as it is, the time required until the water comes out from the reverse surface of the test piece at three places is measured (to an accuracy of 0.5 s). In the test result, the given hydraulic pressure (kPa) shall be stated. If the water does

not come out at three places, the time required until the water comes out at one place or two places is measured or the measurement is made by changing the hydraulic pressure higher. The change of the hydraulic pressure shall be stated in the report.

The extremely small waterdrops which do not grow after being formed shall be ignored.

2.2) **Water leakage method** After applying a given hydraulic pressure, the water which has passed through the test piece is collected in a measuring cylinder after the given time and its volume (ml) is measured to be expressed down to one decimal place per unit area (cm^2). In the test result, the given hydraulic pressure (kPa) and the given time shall be stated.

3.2 Rain test (Shower test) method B

Remarks: In this test, the resistance to surface wetting of test piece can be obtained in accordance with **6.2** of the body.

a) **Apparatus and material** The following apparatus and material shall be used.

1) **Apparatus for artificial rain test** The apparatus shown in Fig. 6 of the body.

2) **Rain quantity gauge** Capable of measuring the quantity of rain.

3) **Filter paper** Use circular filter paper (15 cm in diameter) of class 2 specified in **JIS P 3801**.

4) **Carpet** Capable of preventing water from splashing at the time of rain falling.

5) **Balance** Capable of weighing to an accuracy of 0.1 g.

6) **Water** Distilled or ion-exchange water maintained at (20±2) ℃ in the test shall be used and the chosen alternative shall be stated in the test report.

b) **Procedure** Using the apparatus for artificial rain fall test shown in Annex 1 Fig. 1, select the amount of water supply of the artificial rain fall apparatus and the position where the test piece hold is set so that, when the rain falls on the rain gauge placed on the carpet, the amount of the rain becomes 6 mm/h to 8 mm/h ([3]).

From the specimens specified in 5, sample three sheets of the test pieces each measuring approximately 20 cm × 20 cm, attach one sheet of the filter papers for water absorption of the known mass weighed to an accuracy of 0.1 g to the reverse side of each test piece to mount the test piece on the test piece holder, set them in the place where the artificial rain fall apparatus is put and allow the rain to fall for five minutes ([4]). When the rain falls, immediately take the filter paper away to weigh its mass, calculate the permeation quantity ([5]) by the following formula and express the average value of three measurements down to two decimal places. The chosen alternative in the amount of rain fall and the time of rain fall shall be stated in the test report.

$$\text{Permeation quantity (g)} = M - M_0$$

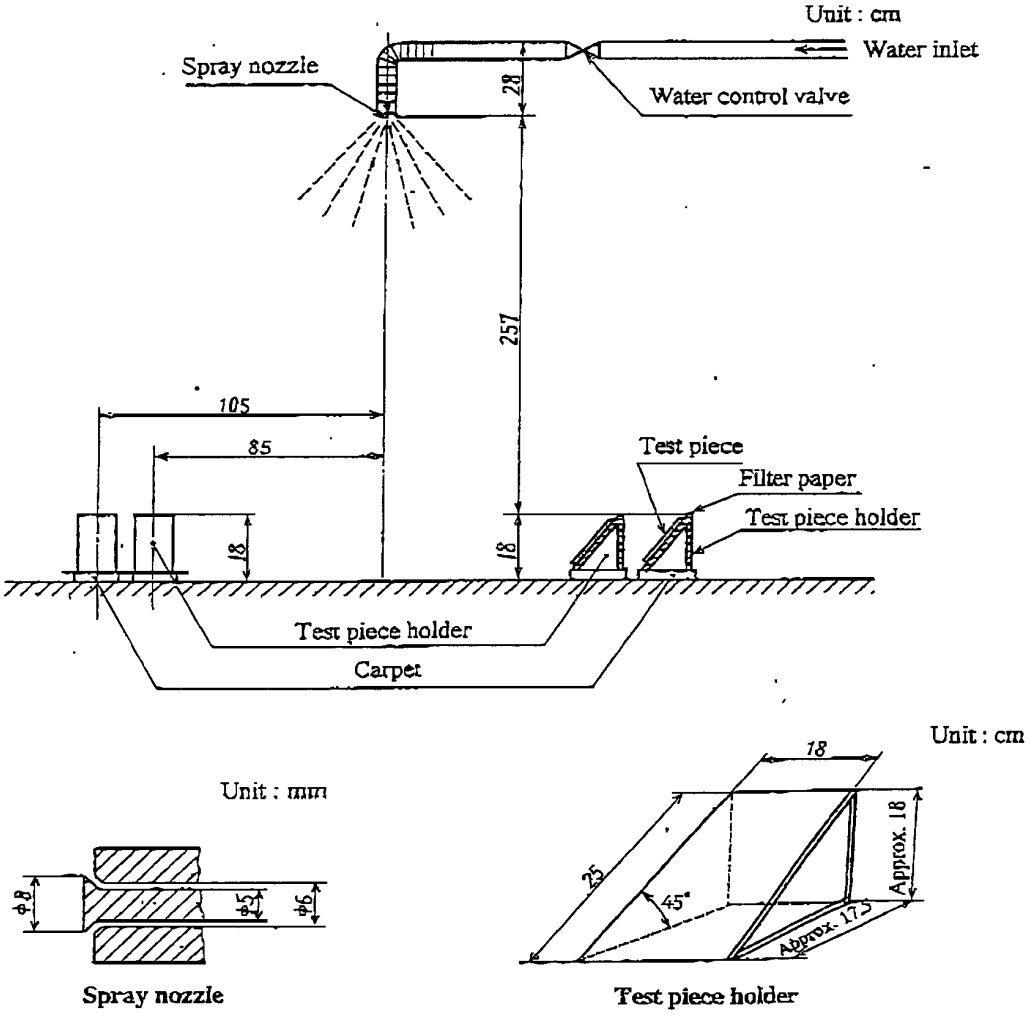
where, M_0: mass of filter paper before the test (g)

M: mass of filter paper after the test (g)

Notes ([3]) The amount of rainfall of 6 mm/h to 8 mm/h is the amount of ordinary rainfall. When testing in an area of heavy rainfall, increase the amount of rain to 10 mm/h to 15 mm/h.

([4]) When testing umbrella cloth, etc., increase the time of rainfall to 15 minutes.

([5]) When the permeation quantity comes to 5 g or more, stop the test and retest by changing the amount of rainfall and the time of rainfall.



Annex 1 Fig. 1 Apparatus for artificial rain fall test

4 Test report The test report shall include the following information for each type of the tests. If the conditioning of specimens specified in 5.2 of the body is carried out, the method of treatment and the number of times of the treatment carried out and the condition at the time of treatment shall be recorded.

a) **Test for water penetration**

1) Type of test

2) Measured values

Water level (Method A) or hydraulic pressure (Method B) at the time of water flow from three places

b) **Rain test (Shower test) method B**

1) Permeation quantity

2) As required, amount of rainfall, time of rainfall and number of times of rainfall

Annex 2 (informative) Determination of waterproofing agents

1 Scope This Annex specifies the determination of waterproofing agents used on waterproofed fabrics by infrared spectrophotometric analysis.

Remarks: Waterproofing agents to be subjected to the determination as given in this Annex are shown in Annex 2 Table 1. Depending on the operation of this Annex, processing agents used for the purposes other than waterproofing may also be detected.

Annex 2 Table 1 Classification of waterproofing agents and extraction solvents

In the Table, ○ indicates capable of extraction, △ indicates capable of extraction depending on the kind of fiber, and × indicates incapable of extraction.

Extraction solvent	Processing agents for non-aeration waterproofing (for coating work)						Processing agents for aeration waterproofing (for water repellent finishing)					
	Rubbers (1)	Polyvinylidene chlorides	Polyvinyl chlorides	Polyethylenes	Polyvinyl acetates	Polyurethanes	Paraffins	Ethylene ureas	Melamines	Methylolamides	Silicons	Fluorines
Methanol	×	×	×	×	○	△	△	○	○	○	△	×
Benzene (2)	○	○	○	○	○	○	○	×	×	×	○	△
Trichlorotrifluoroethane	×	×	△	×	△	×	△	×	×	△	△	○

Notes (1) Types of rubber include isoprenes, butadianes, styrenebutadienes, chloroprenes, acrylonitrile-butadienes, and natural rubbers (isoprene).

(2) When extracting by benzene, acetate fibers are dissolved and interfere with the test, and shall be, therefore, separated by column chromatography, etc.

2 Apparatus An infrared spectrophotometer satisfying the performance requirements specified in **JIS K 0117** shall be used.

3 Procedure

3.1 Extraction Prepare a test piece of approximately 5 g from the specimen, place it in a 200 ml round bottom flask with a reflux condenser, and add 150 ml of extraction solvent (3).

Extract for 1 h by heating to the boiling point of the extraction solvent, and filter the extracted liquid while warm using a sintered-glass filter (4). Put into a 200 ml egg-apple type flask, and concentrate using a rotary evaporator until the extracted liquid becomes 2 ml to 3 ml.

Notes (3) For extraction solvent, use benzene as specified in **JIS K 8858**, methanol as specified in **JIS K 8891**, or 1, 1, 2-trichloro-1, 2, 2-trifluoroethane (refer to the Annex 2 Table 1).

(4) The sintered-glass filter is as given in **JIS R 3503**, and the glass filter plate is that of fine pore symbol 2.

3.2 Refining Fill glass wool into a column chromatography tube of 10 mm to 15 mm in inside diameter and 200 mm to 300 mm in length, and fill the mixture of hexane (6) in column chromatography silica gel (5) until it reaches approximately 150 mm in height. Add in this the extracted liquid concentrated as given in **3.1**, and titrate approximately 100 ml of hexane (6), chloroform (7), and methanol, in that order, into the column chromatography tube. Receive the liquid passed through the chromatography tube for each solvent in a 100 ml egg-apple type flask (8) and remove by fractionating using a rotary evaporator.

Notes (5) Use silica gel of 40 mesh to 80 mesh. To achieve a height of approximately 15 cm, approximately 5 g of silica gel is required for an inner diameter of 10 mm, and approximately 10 g of silica gel is required for an inner diameter of 15 mm.

(6) Use hexane as specified in **JIS K 8848**.

(7) Use chloroform as specified in **JIS K 8322**.

(8) If qualitative determination as given in **3.3** is difficult, reperform the procedure of **3.1**, and separate the mixture by finely fractionating the same solvent in **3.2**.

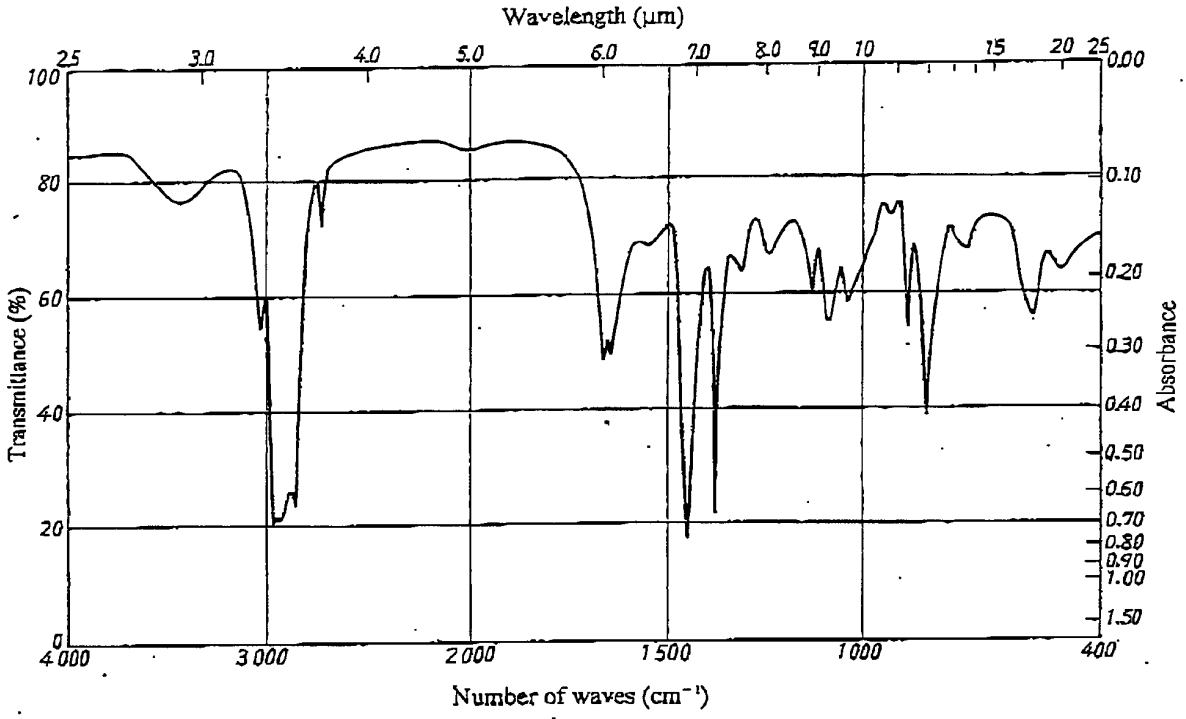
3.3 Determination Prepare the extracts refined in **3.2**, using potassium bromide tablets for solid extract, and using thin film for viscous extract, and measure the infrared absorption spectrum as given in **JIS K 0117**. Determine by comparison with the standard waterproofing agent spectral diagrams previously prepared. Examples of spectral diagrams obtained using the tablet method of potassium bromide are provided in Annex 2 Figs. 1 to 12.

4 Test report The test report shall include the following information:

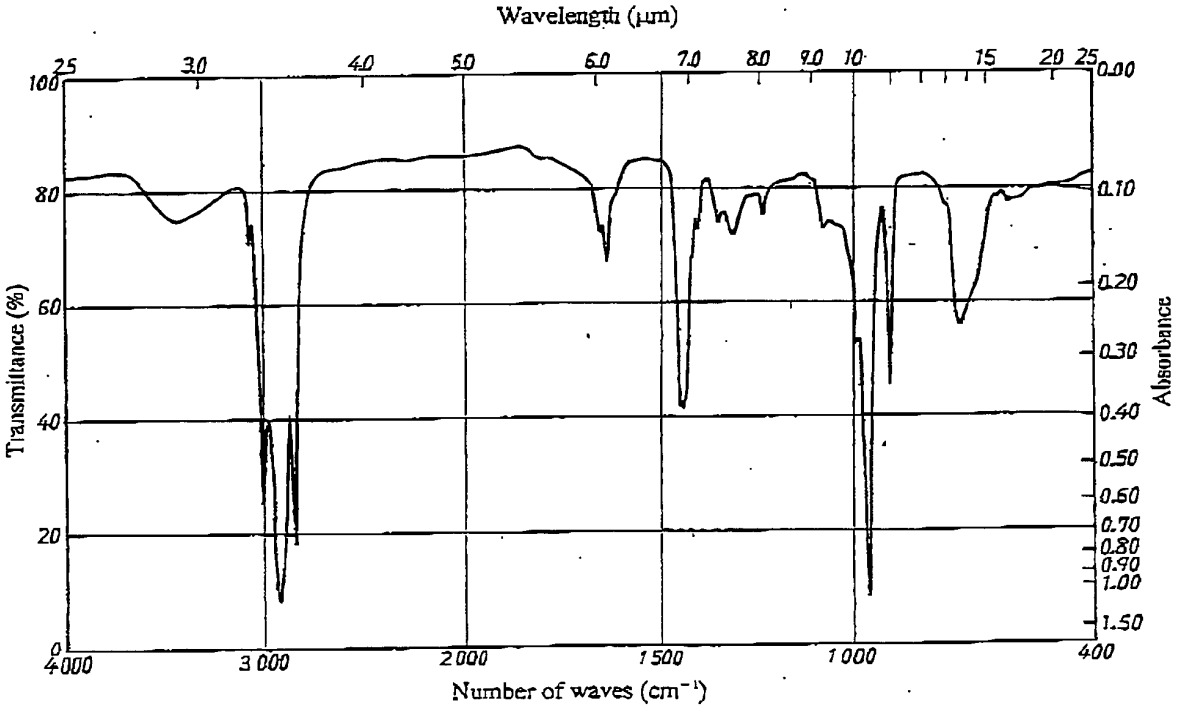
a) Result of determination

b) Extraction solvent

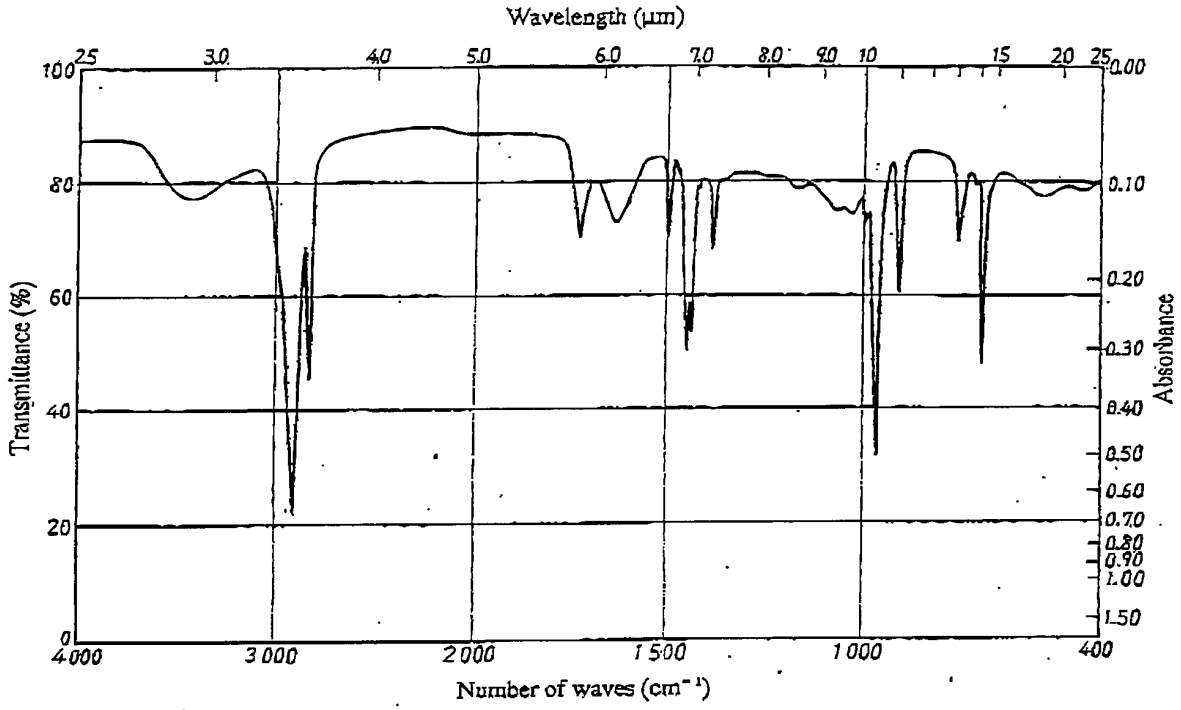
c) Measuring conditions of the infrared absorption spectrum



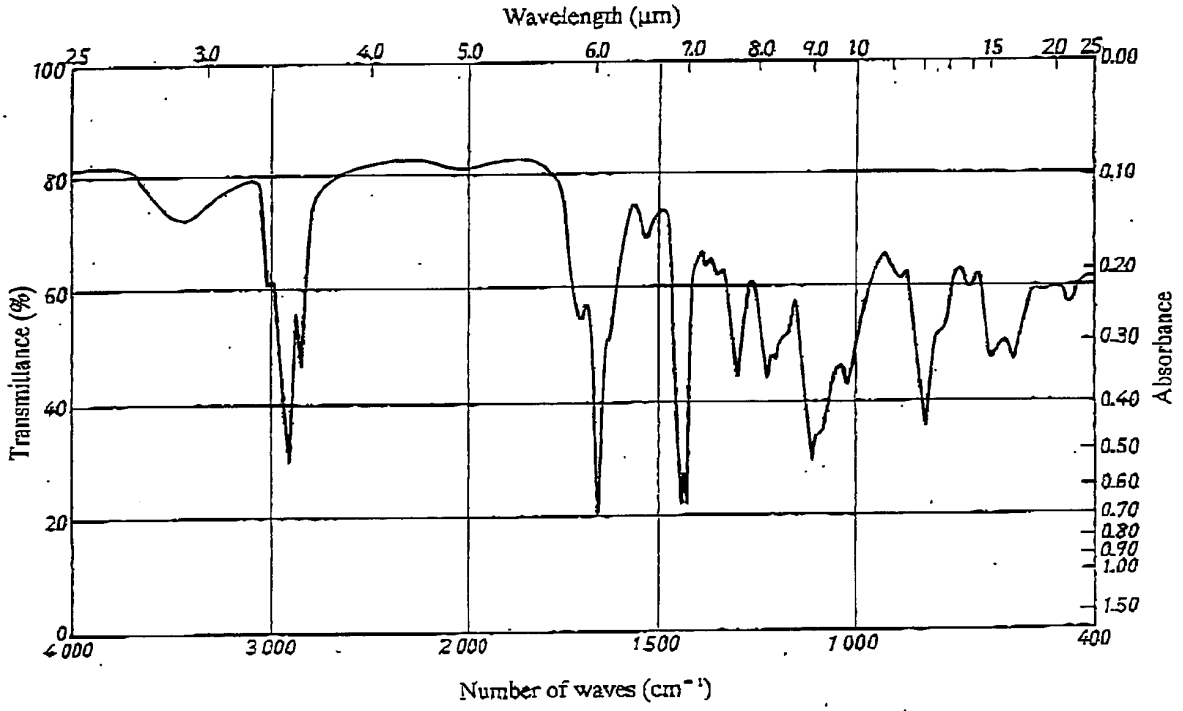
Annex 2 Fig. 1-1 Isoprene rubber



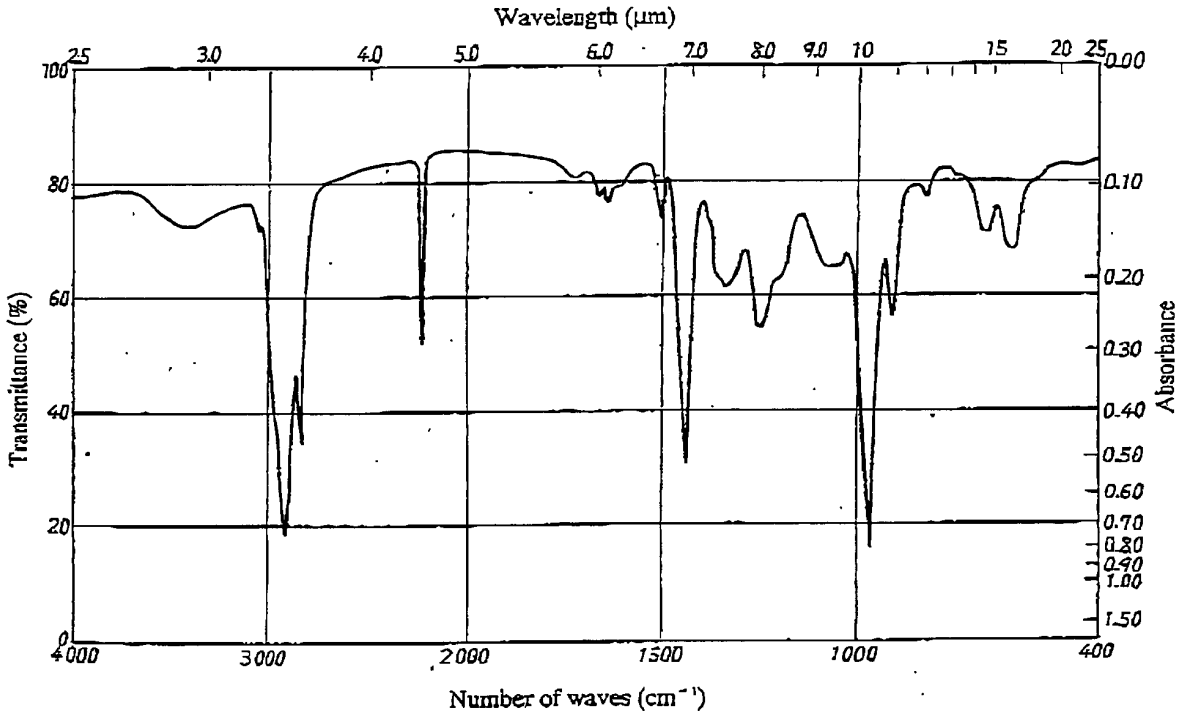
Annex 2 Fig. 1-2 Butadiene rubber



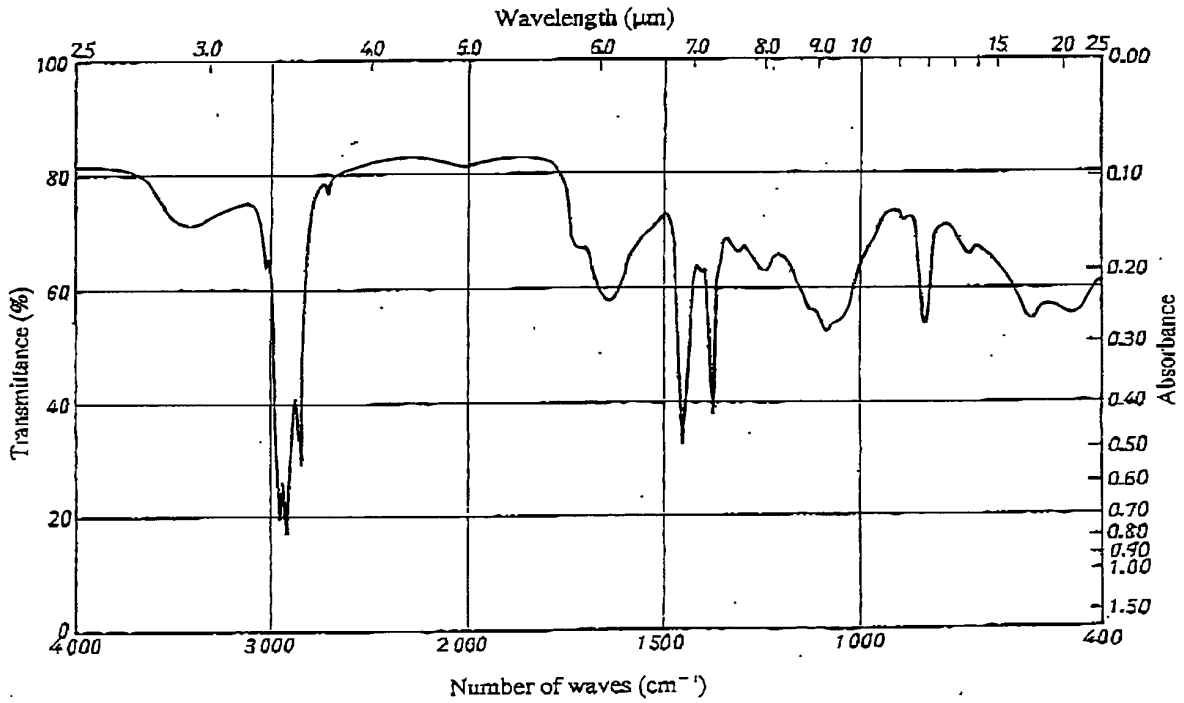
Annex 2 Fig. 1-3 Styrene-butadiene rubber



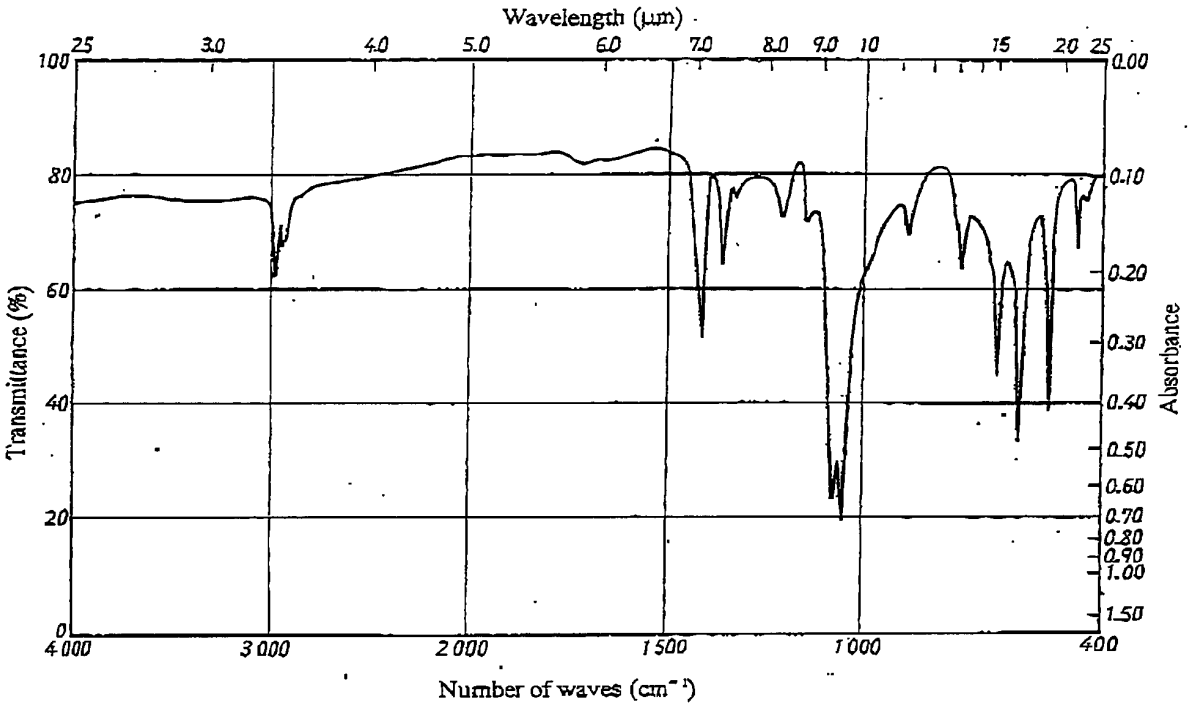
Annex 2 Fig. 1-4 Chloroprene rubber



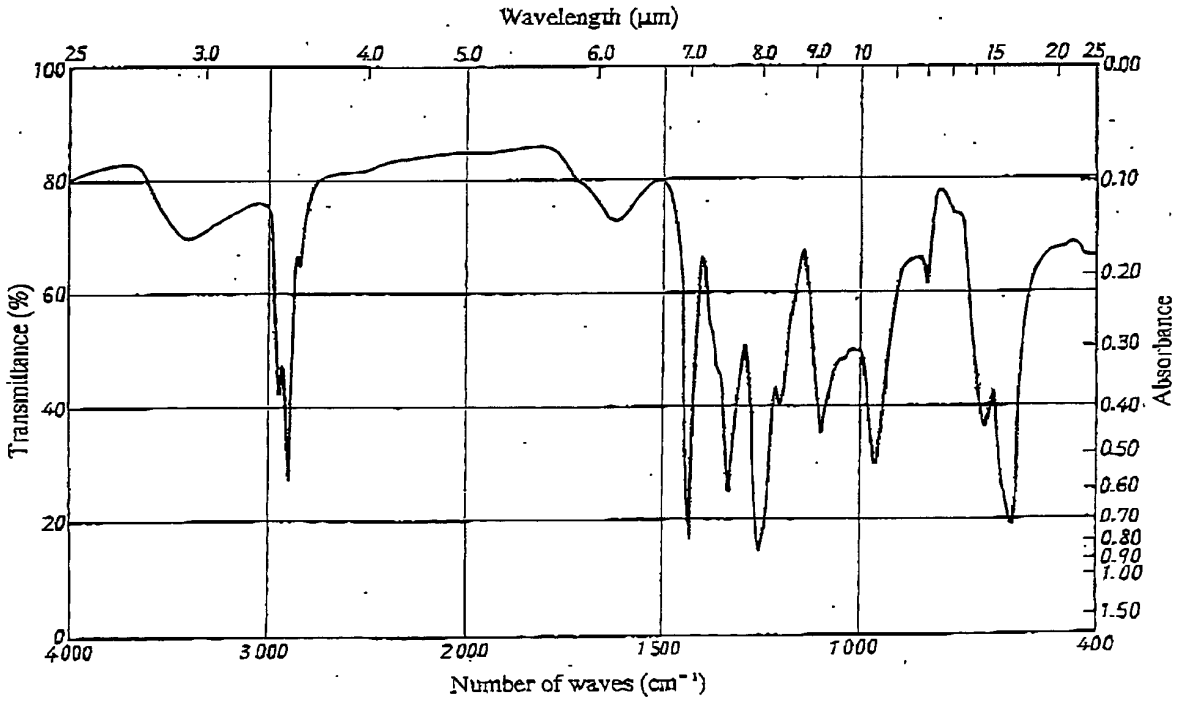
Annex 2 Fig. 1-5 Acrylonitrile-butadiene rubber



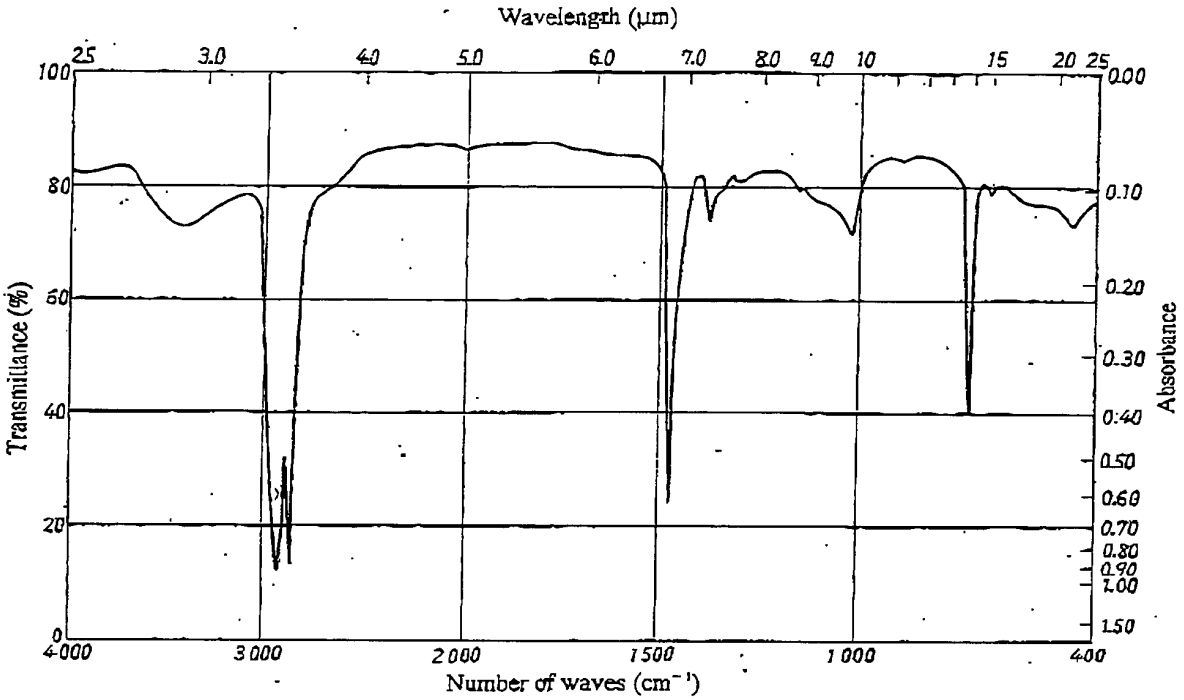
Annex 2 Fig. 1-6 Natural rubber



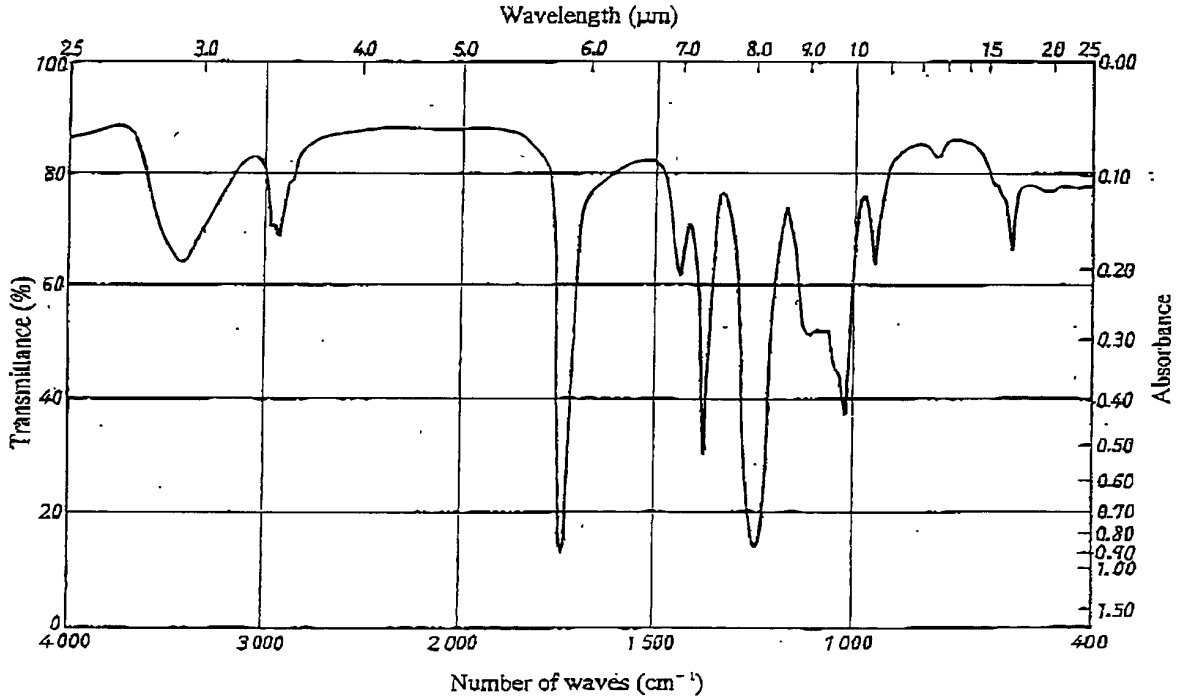
Annex 2 Fig. 2 Polyvinyliden chloride



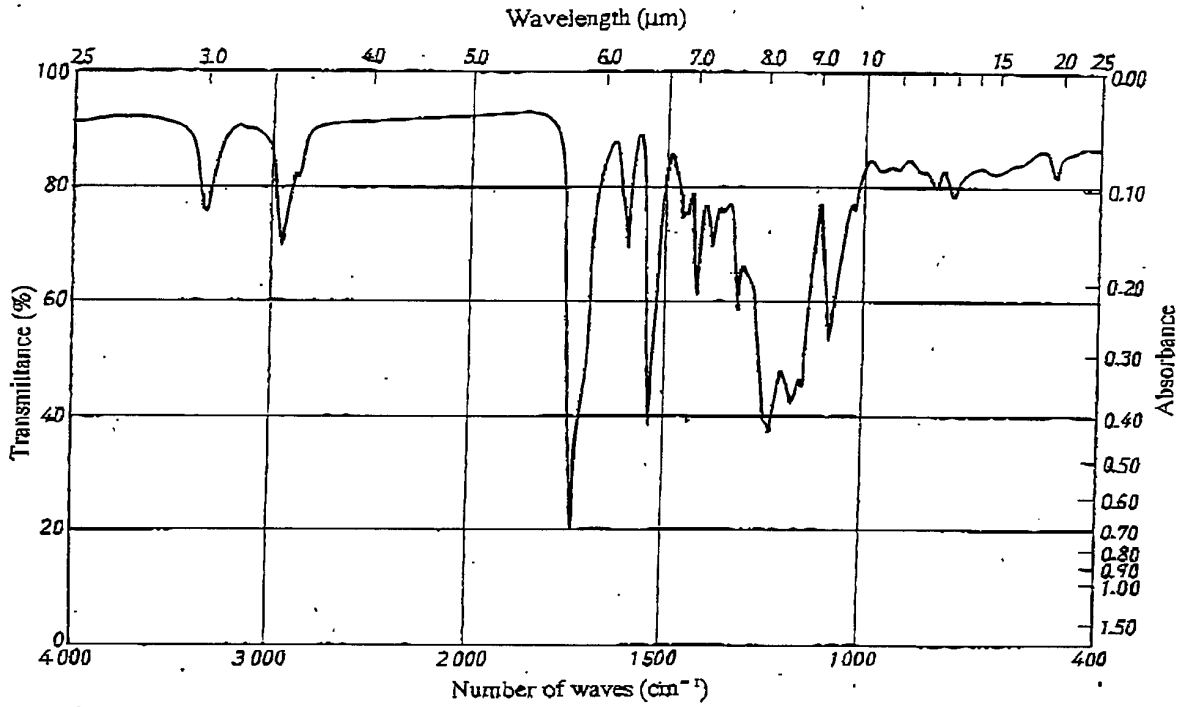
Annex 2 Fig. 3 Polyvinyl chloride



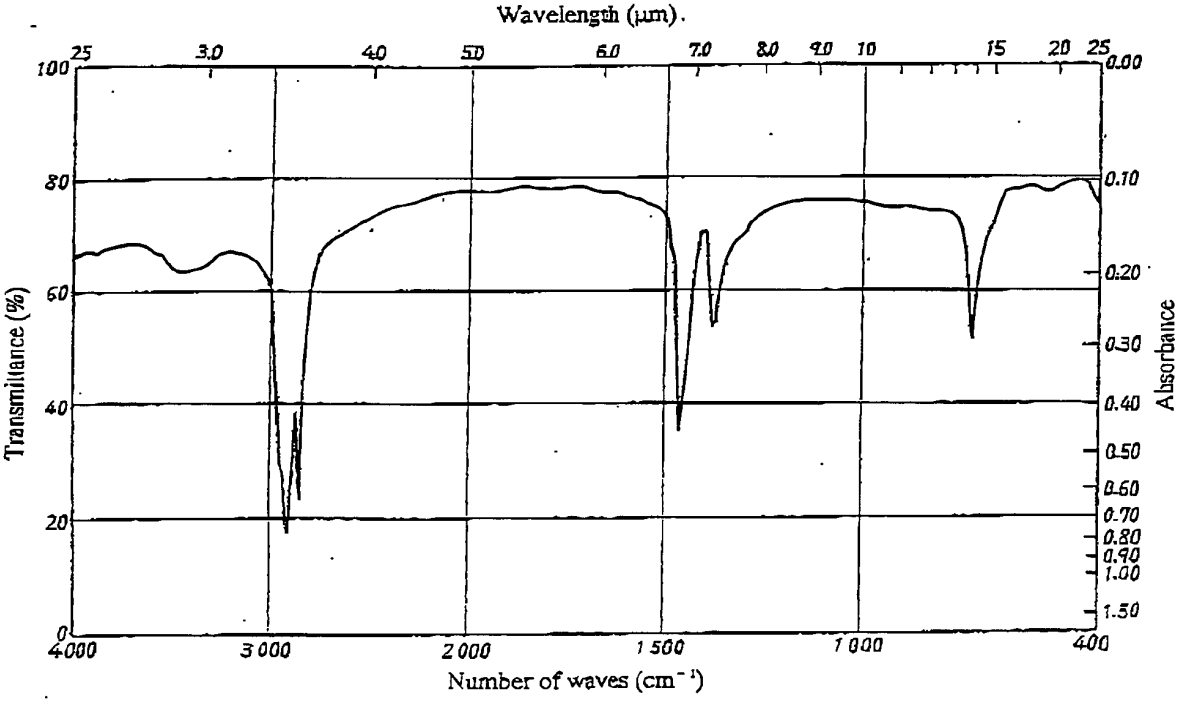
Annex 2 Fig. 4 Polyethylene



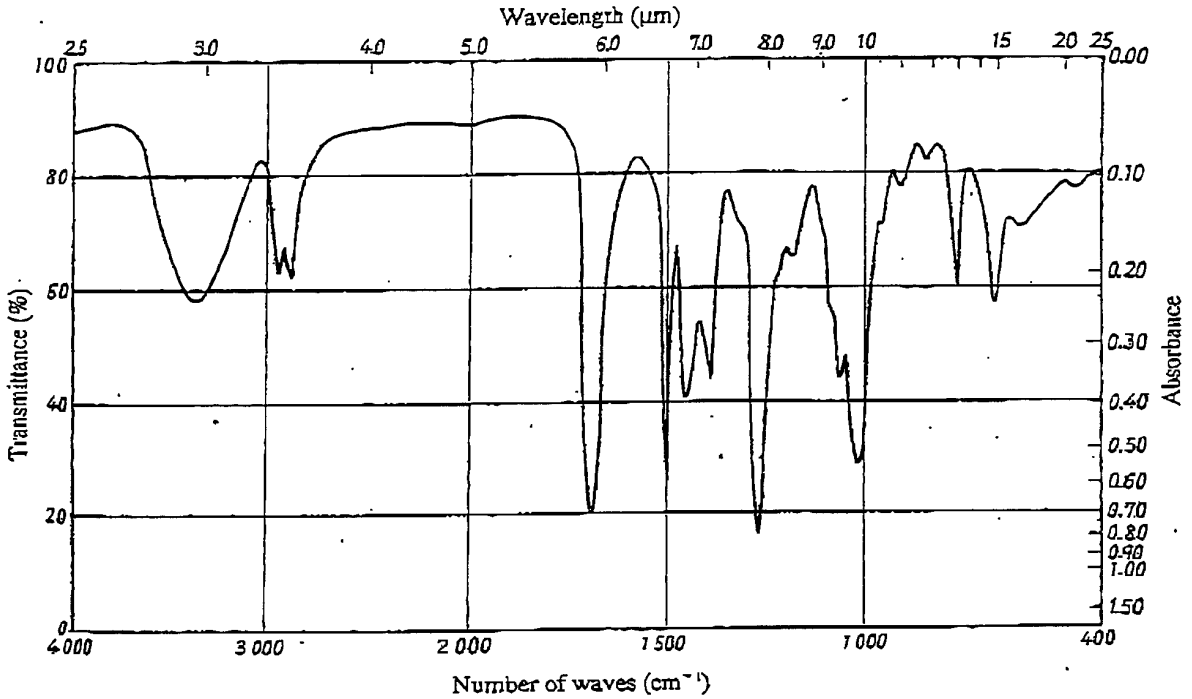
Annex 2 Fig. 5 Polyvinyl acetate



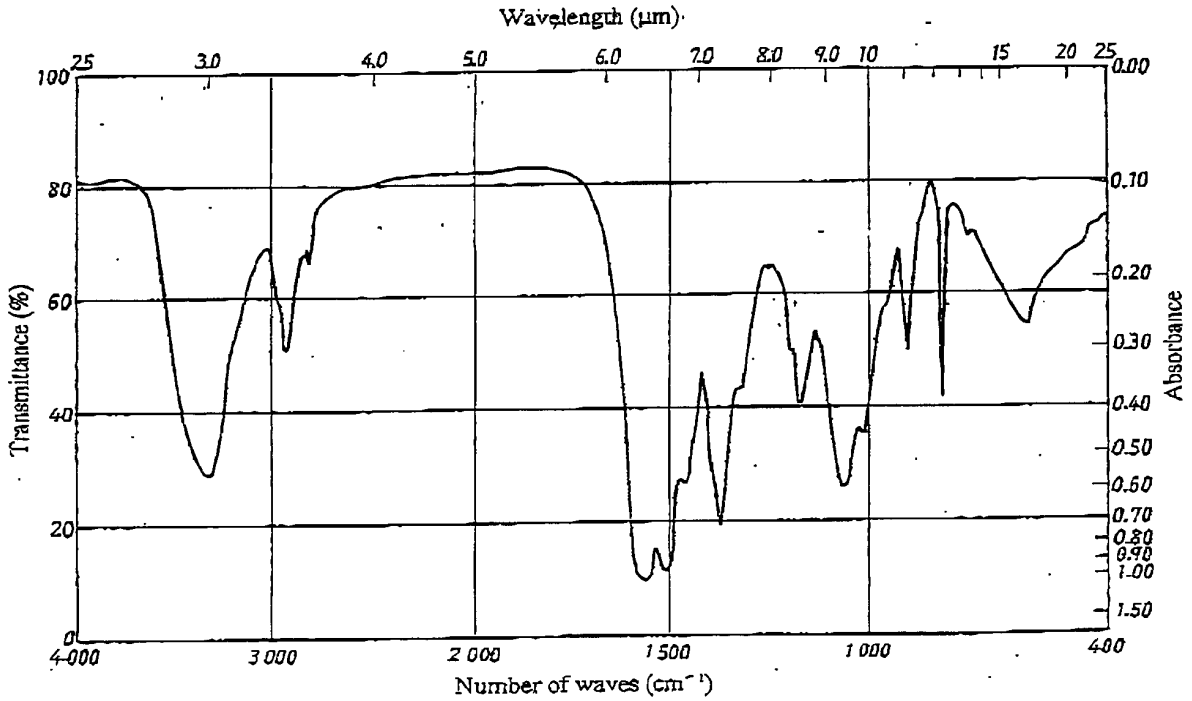
Annex 2 Fig. 6 Polyurethane



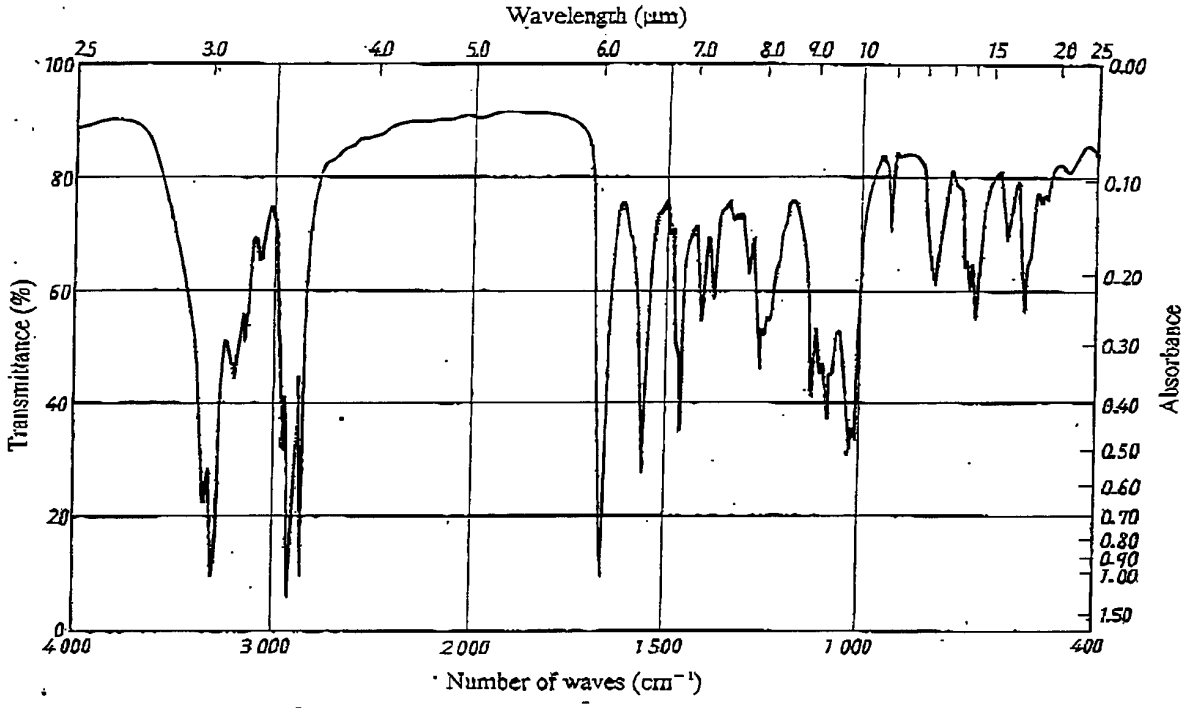
Annex 2 Fig. 7 Paraffin



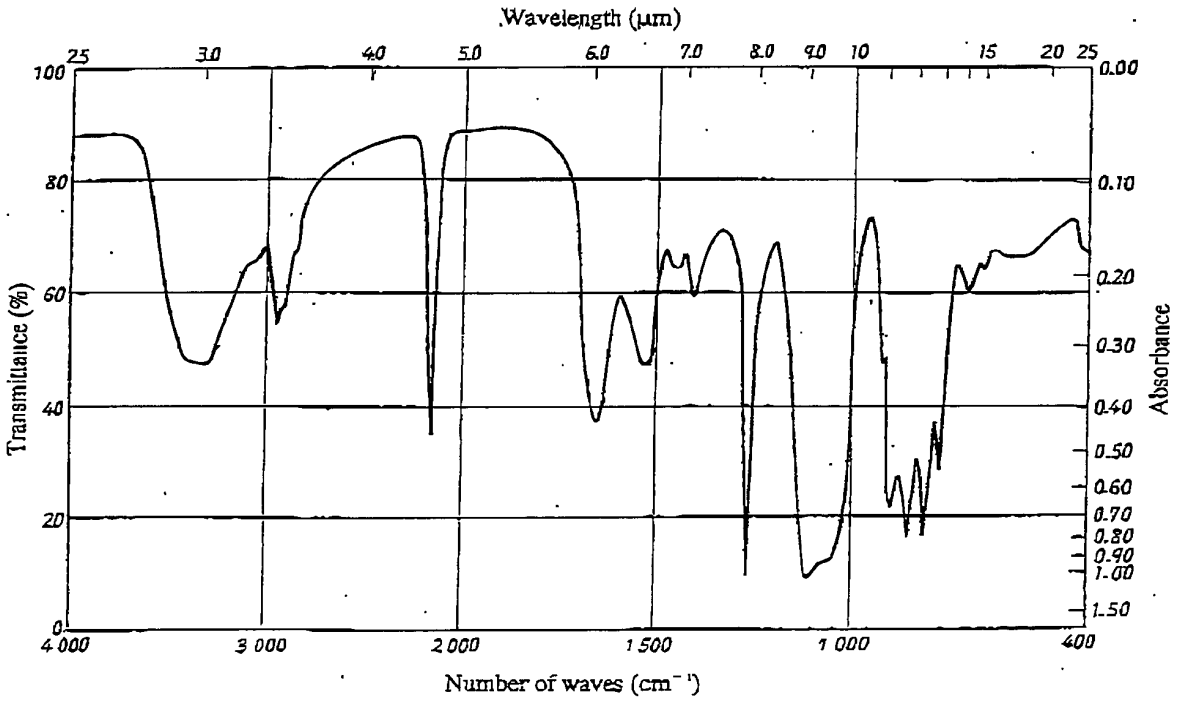
Annex 2 Fig. 8 Ethylene urea



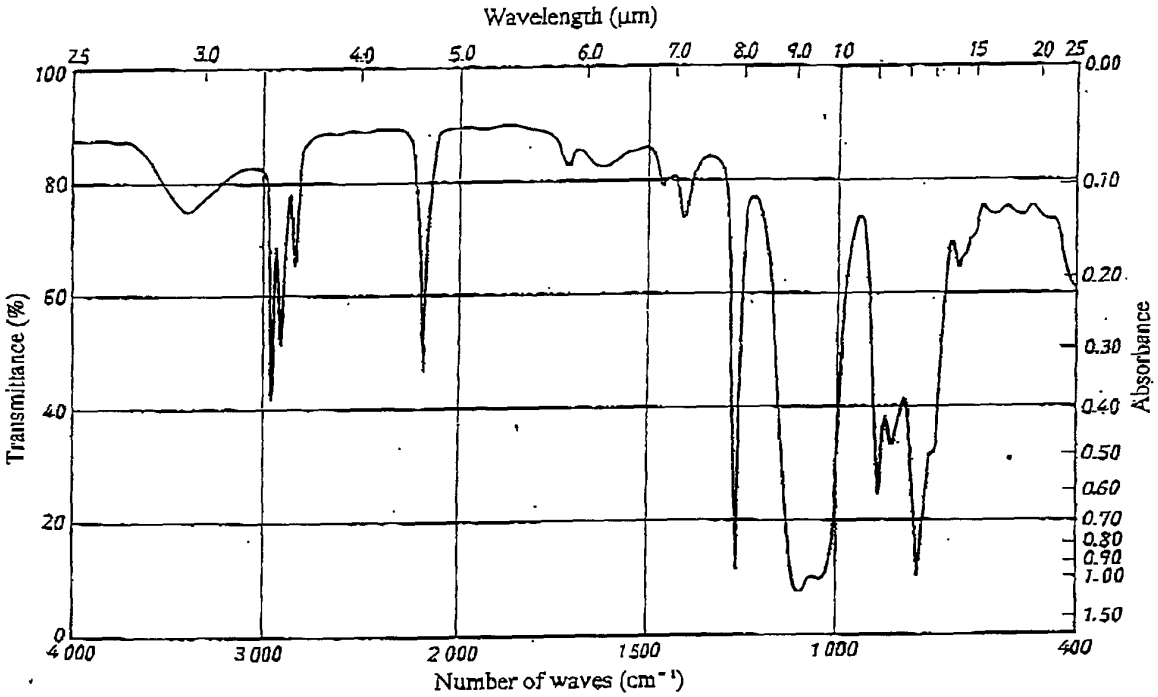
Annex 2 Fig. 9 Melamine



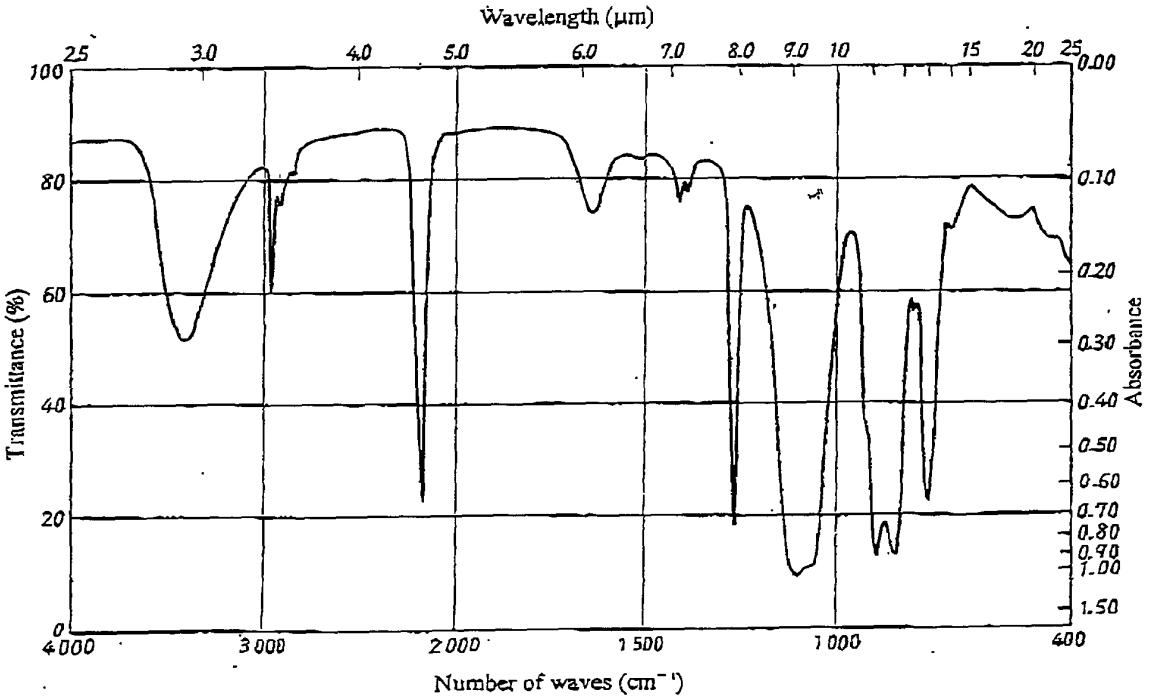
Annex 2 Fig. 10 Methylolamide



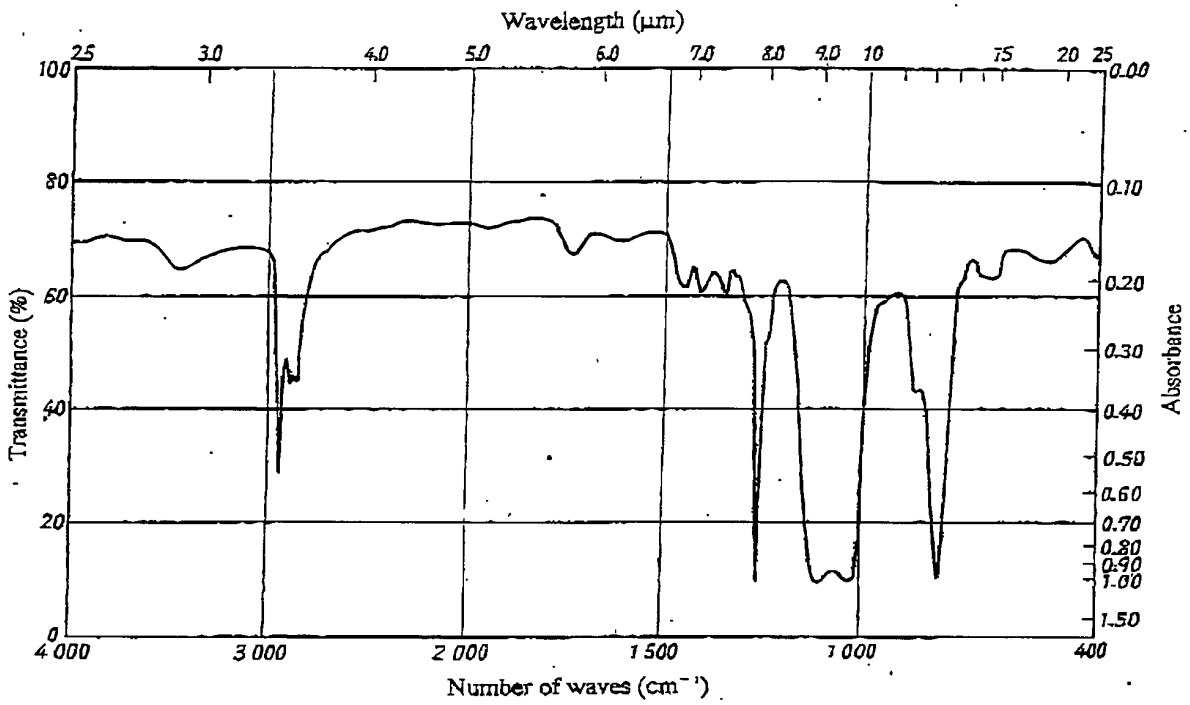
Annex 2 Fig. 11-1 Silicon Part 1



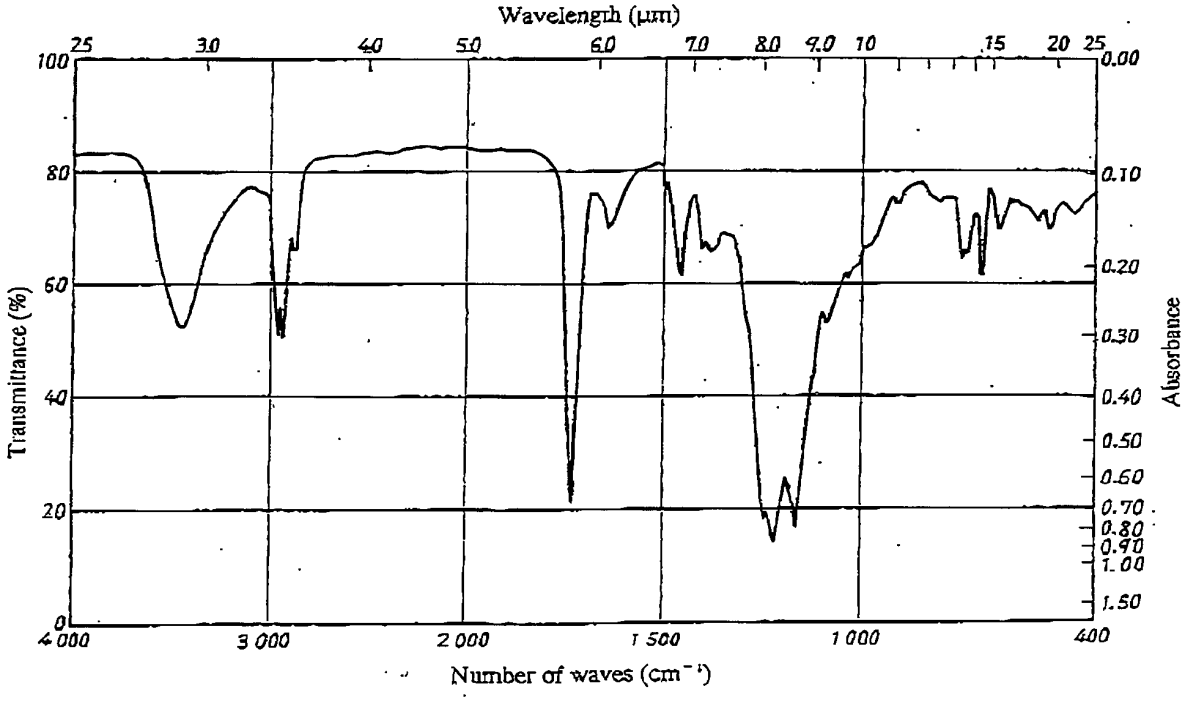
Annex 2 Fig. 11-2 Silicon Part 2



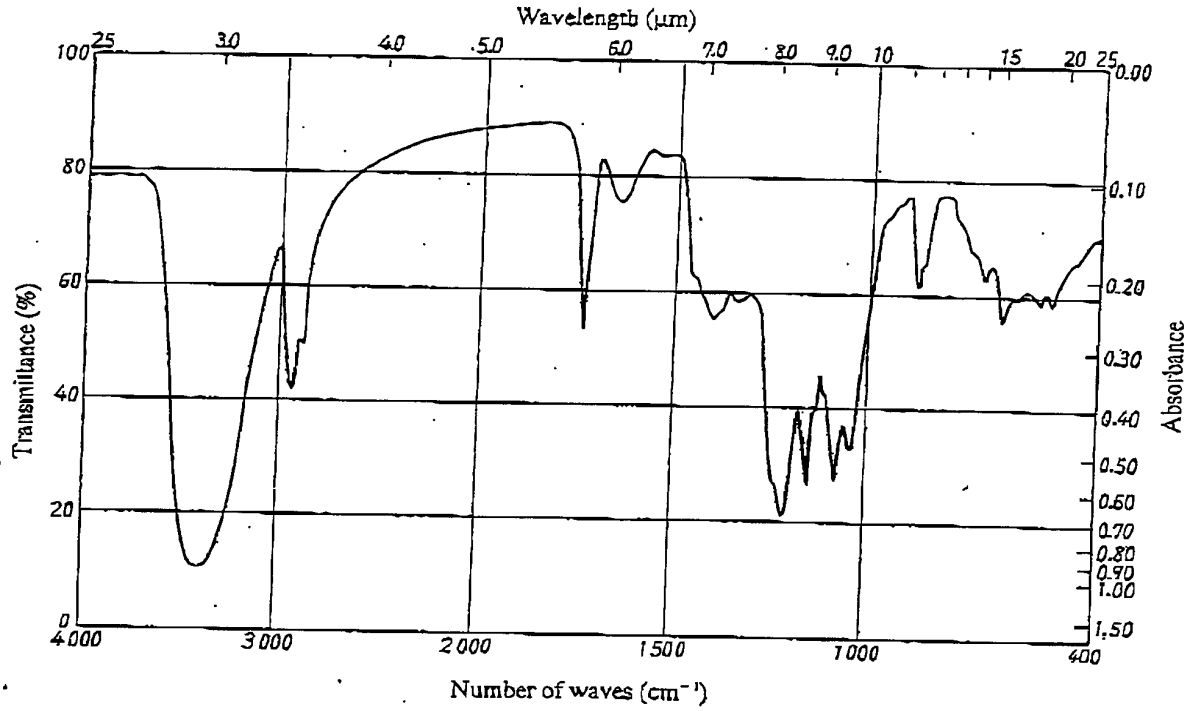
Annex 2 Fig. 11-3 Silicon Part 3



Annex 2 Fig. 11-4 Silicon Part 4



Annex 2 Fig. 12-1 Fluorine Part 1



Annex 2 Fig. 12-2 Fluorine Part 2

Annex 3 (informative) Complement of the body and Annex 1

This Annex 3 is to complement the matters related to the specifications in the body and the Annex 1 thereto and does not form a part of this Standard.

Test methods In addition to the test methods specified in the body, there are the following test methods:

1 **Test for resistance to surface wetting (Water drop method)** In addition to the test for resistance to surface wetting (Spray test) in 6.2 of the body, there is the following water drop method:

a) **Apparatus and material** The apparatus and material are as follows:

1) **Test piece holder** The test piece holder shall be made of metal and be of 15 cm in diameter.

2) **Burette** A burette capable of dividing to 0.1 ml.

3) **Stopwatch** A stopwatch with a 0.5 s scale.

4) **Water** Use distilled water or ion-exchange water, with the temperature at the time of test, as a rule, (20±2) ℃. If the temperature is other than this, record the temperature.

b) **Procedure** From the specimen specified in 5 of the body, prepare three test pieces of approximately 20 cm × 20 cm, attach to the test piece holder of 15 cm in diameter so that there are no wrinkles, drop 0.1 ml water on each of five places from a height of 2 cm above the flat test piece using a burette, measure the time (s) until the water penetrates into the test piece, and express as the mean value of the three test pieces to one decimal place.

2 **Rain test (Shower test)** In addition to the rain tests in 6.3 of the body and 3.2 of Annex 1, there are the following Methods A and B. Method A is very similar to **AATCC** Test Method 35 Water Resistance: Rain Test. Method B is very similar to **BS 5066**, Method of test for the resistance of fabrics to an artificial shower.

a) **Method A (AATCC Method)**

1) **Apparatus and material** The apparatus and material are as follows:

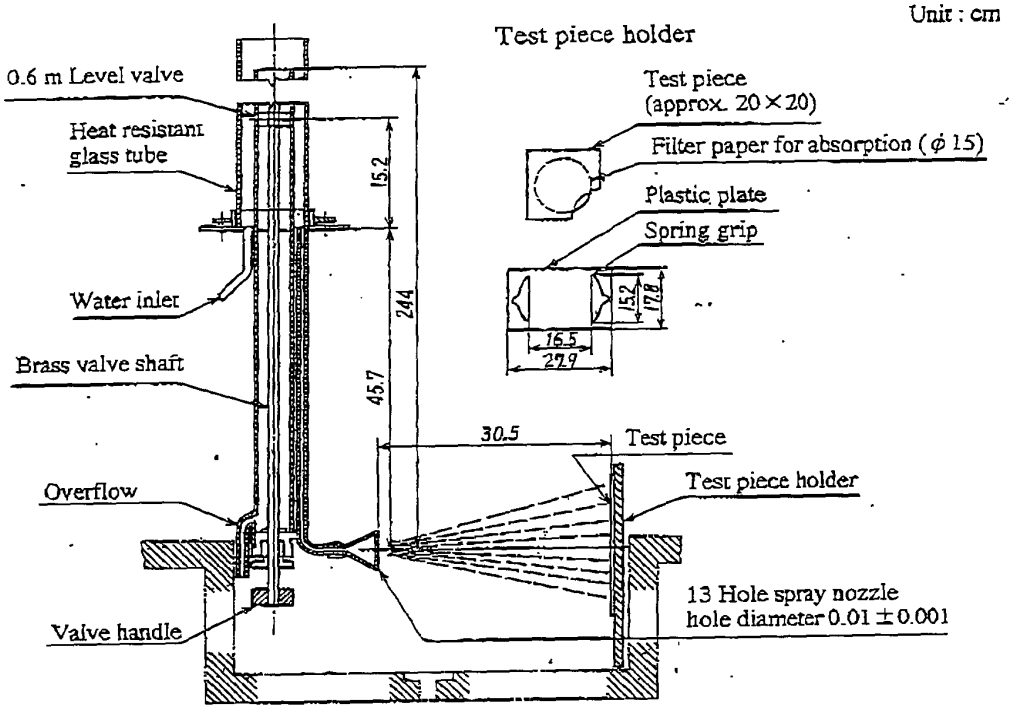
1.1) **Water tester** The water tester is as shown in Annex 3 Fig. 1, and is capable of raising the height of the water column between 0.6 m and 2.4 m, by 0.3 m as a unit.

1.2) **Filter paper for absorption** Use class 2 circular filter paper (15 cm in diameter) as given in **JIS P 3801**.

1.3) **Balance** A balance capable of measuring to the nearest 0.1 g.

1.4) **Water** Use distilled water or ion-exchange water, with the temperature at the time of test, (20±2) ℃. If a different temperature is used, record the temperature.

Unit : cm



Annex 3 Fig. 1 Rain tester

2) **Procedure** From the specimens of 5 of the body, prepare three test pieces of approximately 20 cm × 20 cm (1), press one sheet of 15 cm diameter filter paper for absorption weighed to the nearest 0.1 g tightly to the back of each test piece, fix them to the test piece holder, and attach to the rain tester so that the center of the test piece surface coincides with that of the spray nozzle with 30.5 cm in between. Adjust the height of the water column (2), make the spray nozzle horizontal, supply water from the water inlet, spray from the nozzle for 5 min, immediately weigh by blotting filter paper, calculate the permeation quantity (g) using the following formula, and express the mean value of the three test pieces to one decimal place. Record the height of the water column (cm) at the time of test in the test results.

$$\text{Permeation quantity (g)} = M - M_0$$

where, M_0: mass of the filter paper before the test (g)

M: mass of the filter paper after the test (g)

Notes (1) The test piece may be prepared with a single sheet, double sheets, or material of surface cloth and lining put together as used for raincoats, etc.

(2) Set the height of the water column between 0.6 m and 2.4 m, with a unit of 0.3 m. As required, increase the height of the water column by the unit of 0.3 m from the maximum height where the permeation quantity becomes 0 g until the minimum height where the filter paper is torn or the permeation quantity exceeds 5 g, and determine the permeation quantities at the respective heights of the water column.

b) **Method B (BS Method)** Method B (BS Method) is also called the WIRA method, and is as follows:

1) **Apparatus and material** The apparatus and material are as follows:

1.1) **Shower tester** The shower tester is as shown in Annex 3 Fig. 2. For the shower head, use perforated polytetrafluoroethylene plate as shown in Annex 3 Fig. 3 and for test piece holder, use a ribbed glass receiving plate (81 mm × 135 mm) as shown in Annex 3 Fig. 4

1.2) **Vibrator** The vibrator is such that the test piece holder freely drops 30 cm vertically along the column standing on a heavy stand and strikes a rubber stopper to give an impact to the test piece.

1.3) **Stopwatch** A stopwatch with a 0.5 s scale.

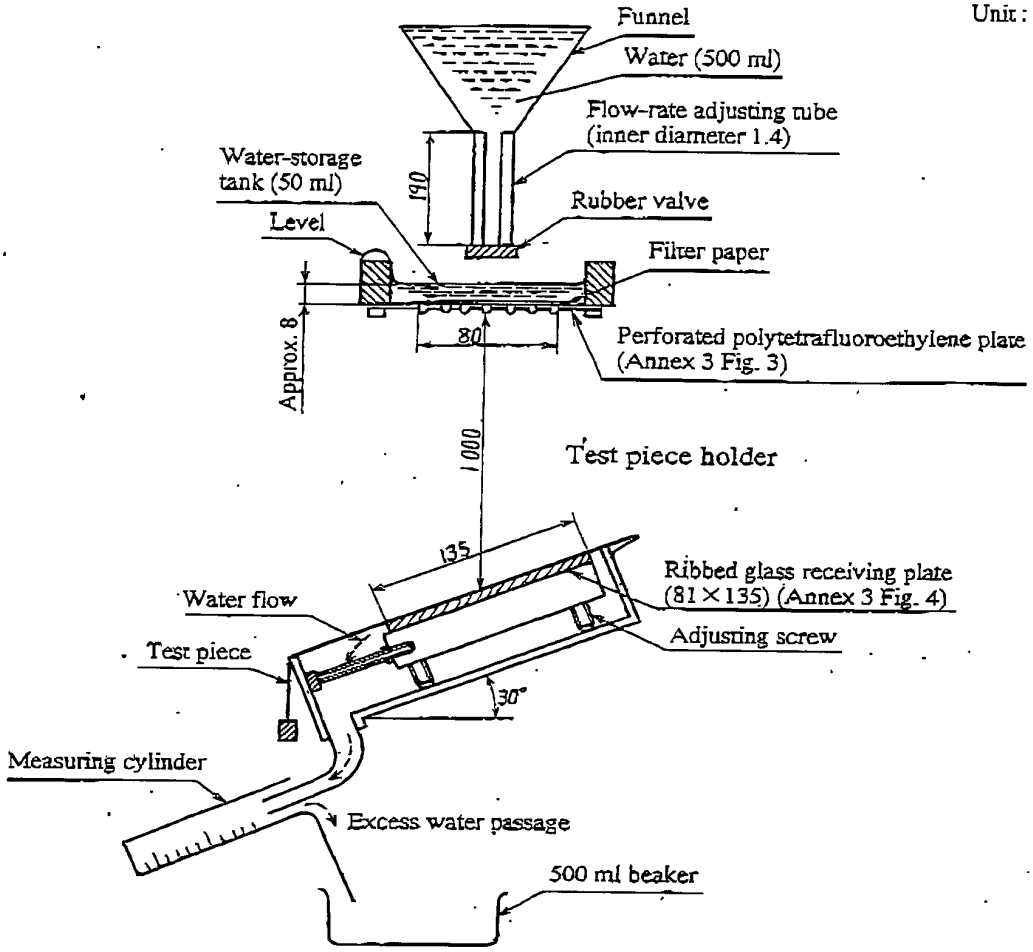
1.4) **Balance** A balance capable of measuring to the nearest 1 mg.

1.5) **Measuring cylinders** Measuring cylinders of 50 ml and 500 ml to pour water into the shower head, and a 10 ml to 500 ml cylinder to measure the quantity of water passing through a test piece.

1.6) **Filter paper** Class 2 filter paper as given in **JIS P 3801**, circular in shape and 90 mm in diameter.

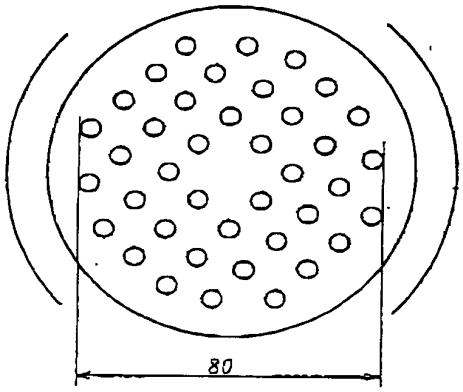
1.7) **Water** Use distilled water or ion-exchange water, with the temperature at the time of test, as a rule, (20±2) ℃. If the temperature is other than this, record the temperature.

Unit : mm



Annex 3 Fig. 2 Shower tester

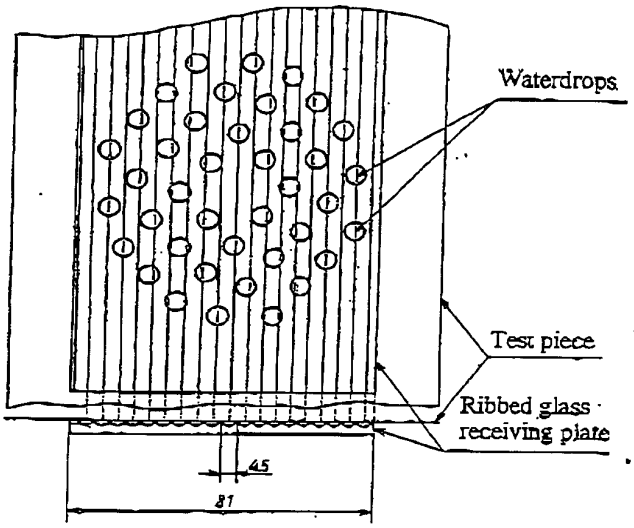
Unit : mm



(Perforated polytetrafluoroethylene plate which has 40 holes of 4 mm in diameter within a circular area of approximately 80 mm in diameter.)

Annex 3 Fig. 3 Shower head

Unit : mm



Annex 3 Fig. 4 Where shower waterdrops fall on the ribbed glass receiving plate

2) **Test preparation** Wash the ribbed glass receiving plate with water (3), immerse it in a 2 *l* beaker containing approximately 1.5 *l* of a surface-active agent washing liquid, let stand for 15 min, wash with running water (3) for 10 min, further immerse in a 2 *l* beaker containing water at approximately 50 ℃ for 1 min, take it out and dry.

For the shower head part, dry the perforated polytetrafluoroethylene plate using tissue paper, place 90 mm diameter filter paper on top, add 2 ml to 3 ml water to let the plate adhere tightly to the filter paper, further press the filter paper slightly into the holes of the plate, and attach them to the bottom of water storage tank.

Make the water storage tank horizontal using a level, add 50 ml of water, and raise the water level to approximately 8 mm. The water drops pass uniformly from each hole through the filter paper (4). Within 2 s to 3 s the water drops stop falling. Confirm that the water level at that time is approximately 5 mm.

From the specimens of 5 of the body, prepare four test pieces of 125 mm × 250 mm. Weigh the mass to the nearest mg, place on the ribbed glass receiving plate, attach to the test piece holder as shown in Annex 3 Fig. 2, fix at the proper place on the tester, and connect the tester to the measuring cylinder and the beaker.

Notes (3) The water specified in 1) 1.7) need not be used.

(4) If the water drops do not fall uniformly from the holes of the shower head, adjust the position of the filter paper with a glass rod or replace the filter paper.

3) **Procedure** Using the shower tester given in Annex 3 Fig. 2, pour 500 ml of water into the glass funnel, open the valve under the flow-rate adjusting tube, and spray over the test piece for approximately 7.5 min (5). 8.5 min $^{+20}_{0}$ s later, detach the test piece, use the vibrator to remove the excess water-drops, and immediately weigh the mass to the nearest 1 mg (6). Calculate the water absorption (%) of the test piece using the

following formula, and measure the volume of water received by the measuring cylinder after passing through the test piece, as shown in Annex 3 Fig. 2, which is taken as the water leakage (ml) ([7]), and express the water absorption and water leakage as the mean value of the four test pieces ([8]).

$$\text{Water absorption } (\%) = \frac{M - M_0}{M_0}$$

where, M_0: mass of the test piece before the test (mg)

M: mass of the test piece after the test (mg)

Notes ([5]) Adjust the flow-rate adjusting tube so that 500 ml water drops in 7.5 min ± 10 s. Open the valve so that the water in the funnel maintains the water level in the water storage tank at approximately 7 mm, giving a stable shower flow (at this time, the waterdorps are approximately 65 mg).

([6]) In order to prevent the loss of water from the test piece with water absorbed, weight the test piece in a closed container.

([7]) If necessary, measure the time until the amount of water leakage reaches 10 ml.

([8]) Measure to the nearest 0.5 % if the water absorption is 10 % or less, and to the nearest 1 % if it exceeds 10 %. Measure to the nearest 0.5 ml if the amount of water leakage is 10 ml or less, and to the nearest 1 ml if it exceeds 10 ml.

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EXHIBIT B

UDC 621.798.15:620.198.2

JIS

JAPANESE INDUSTRIAL STANDARD

Testing Methods for Determination of the Water Vapour Transmission Rate of Moisture-Proof Packaging Materials (Dish Method)

JIS Z 0208 - 1976

8 pages

Translated and Published

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Japanese Standards Association

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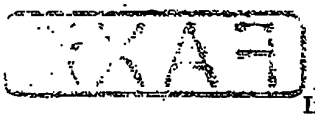


In the event of any doubt arising,
the original Standard in Japanese is to be final authority.

UDC 621.798.15:620.198.2

JAPANESE INDUSTRIAL STANDARD J I S

Testing Methods for Determination of the Water Vapour Transmission Rate of Moisture-Proof Packaging Materials (Dish Method)

Z 0208-1976
(Reaffirmed: 1994)

1. Scope

This Japanese Industrial Standard specifies the method using the water vapour transmission dish for testing the water vapour transmission rate of the moisture-proof packaging materials such as plastic film, converted paper and the like.

2. Definition

The water vapour transmission rate is the quantity of vapour passing through the unit area of filmy substance for the definite hour. In this standard, when constituting the boundary surface by the moisture-proof packaging materials at the temperature of 25°C or 40°C, and keeping the air of one side at a relative humidity of 90 % and the air of the other side at the dry state by moisture absorbent, the value having converted the mass (g) passing through this boundary surface for 24 h into the value per 1 m^2 shall be defined as the water vapour transmission rate.

Because the affects of temperature and humidity on the water vapour transmission rate are not simple, the rate having been estimated from the measured value under the temperature and the humidity condition different from the testing condition as specified in this standard cannot be regarded as the water vapour transmission rate termed in this standard.

3. Apparatus

3.1 Water Vapour Transmission Dish The water vapour transmission dish, hereinafter referred to as the "dish", shall meet the following conditions. An example of the dish and its accessories is shown in Attached Figure.

(1) The area of water vapour transmission shall be not less than 25 cm^2 and be capable of specifying its area clearly. The area of water vapour transmission shall be calculated from the inner diameter of the ring.

(2) The quality of material shall have no permeability for the vapour and produce no corrosion and the like under the testing condition.

(3) The dish shall have enough rigidity not to be transformed during operation.

(4) The peripheral part of test piece shall be sealed completely.

Applicable Standards:

JIS K 8123-Calcium Chloride, Anhydrous

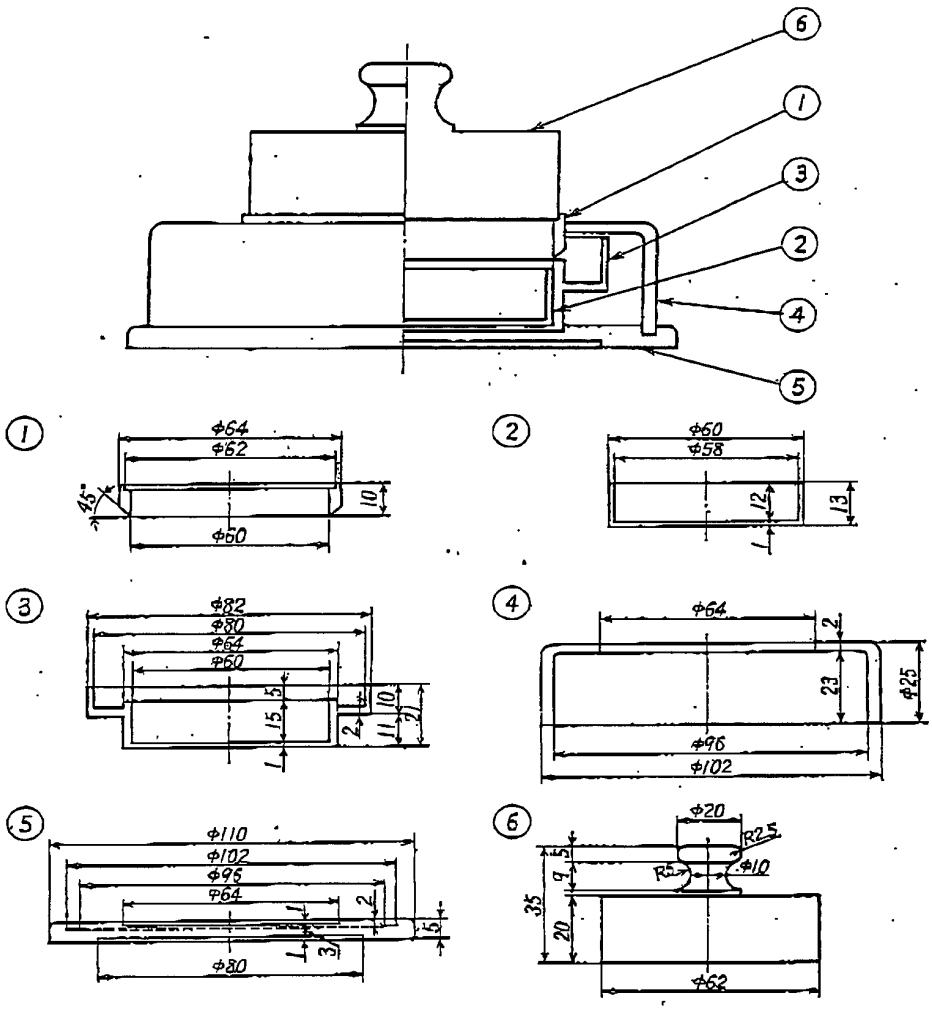
JIS Z 8401-Rules for Rounding off of Numerical Values

JIS Z 8801-Sieves for Testing Purposes

2
Z 0208-1976

Attached Figure An Example of Water Vapour Transmission Dish and Its Accessories

Unit: mm



Number	Name	Remark
1	Ring	Aluminium material, treated by anodic oxidation vapour.
2	Dish	Dish made from glass and others, and of light weight.
3	Cup	Aluminium material, treated by anodic oxidation vapour.
4	Guide	Guide made of brass casting or the like.
5	Cup base	Base made of brass casting or the like.
6	Weight	About 500 g in mass of brass casting or the like.

3.2 Cover Where the use of cover is required [see (10) in 6.], its cover shall be one capable of covering the one side of test piece completely and it is advisable that its material is same as that of cup.

3.3 Thermo-Hygrostat The thermo-hygrostat shall be one in which the air kept at the specified temperature and humidity can circulate at a velosity of 0.5 to 2.5 m/s above the test piece. The temperature and humidity conditions at the time of test shall be as follows:

Condition A Temperature: 25 ± 0.5°C Relative humidity: 90 ± 2 %

Condition B Temperature: 40 ± 0.5°C Relative humidity: 90 ± 2 %

3.4 Chemical Balance The chemical balance shall be capable of weighing the mass of cup to 0.1 mg.

4. Chemical Agents

4.1 Moisture Absorbent The moisture absorbent shall be as specified in JIS K 8123. The absorbent having a grain size passing through the sieve of nominal size 2380 μm as specified in JIS Z 8801 and remaining on the 590 μm sieve shall be used.

4.2 Sealing Waxes Use the sealing waxes meeting the following conditions. Further, it is preferable that the filler and the insoluble solid component are not included.

(1) It shall be difficult to be peeled off and easy to be operated to seal with wax.

(2) It shall be not fragile at room temperature and have no water absorption, nor hygroscopic property, nor fear of oxidation.

(3) It is required that the sealing waxes are not softened and deformed when being exposed under the temperature and humidity condition B and the change in the mass of not less than 1 mg in 24 h is not produced, where its exposed surface area is 50 cm^2.

Remark: The following are examples of compounding (in mass ratio) of sealing waxes.

(a) Microcrystalline wax 60 % and refined crystal paraffin wax 40 %

(b) Paraffine wax 80 % having a melting point of 50 to 52°C and viscous polyisobutylene (one having a low polymerization) 20 %

(c) Mixture of waxes having oil of 1.5 to 3 % at a melting point of 60 to 75°C

5. Test Piece

Take the test piece with sufficient cares to represent its sample, cut off not less than three test pieces, which have circular shape having a diameter larger by about 10 mm than the inner diameter of the cup to be used, from the same sample to offer the test.

Where the discrimination of the both top and bottom sides of test specimen is clear, the direction of the side of test piece can be kept constant according to the use of that material when fitting the test piece with the cup. When measuring on the both sides, prepare not less than three test pieces on each side.

6. Operation

Where the cup as shown in Attached Figure is used, fit the test piece with the cup by the following operation and carry out the test. Where other cup is used, carry out the test operation corresponding thereto.

(1) Cleanse the cup and after having dried it, warm it up to a temperature of about 30 to 40°C.

(2) Put the dish containing moisture absorbent in the cup and place it on the cup base kept horizontally. At this time, keep the surface of moisture absorbent horizontal as far as possible so that the distance from the underside of test piece will become about 3 mm.

(3) Put the test piece on the position to become concentric with the cup.

(4) Cover the guide to fit with the groove of cup base.

(5) Push the ring in as shown in Attached Figure until the test piece will contact closely with the upper edge of cup to fit the guide and put the weight on it.

(6) Draw the guide perpendicularly up with cares not to move the ring to remove it.

(7) While rotating the cup horizontally, flow the melted sealing waxes (1) into the groove at the peripheral part of the cup and seal the edge of the test piece. At this time, take cares not to produce cracks, bubbles and others.

(8) Remove the weight and the cup base after the sealing waxes has been solidified. Clean the sealing waxes that have been stuck to the part except the sealing part (sides and bottom of the cup and others) by the cloth with a suitable solvent soaked to remove and take as test specimen.

(9) Put the test specimen in the thermo-hygrostat kept at the specified test condition.

(10) After having placed the test specimen in the thermo-hygrostat not less than 16 h, take it out of the apparatus, let it keep balance with room temperature and measure the mass by the chemical balance. Where the side exposed to the outside of the test piece is the material having a large hygroscopic property, cover the test specimen immediately after taking it out of the thermo-hygrostat apparatus to lessen the change of moisture content as far as possible (2).

(11) Put the test specimen in the thermo-hygrostat again, take the cup out at a suitable time interval, repeat the weighing operation and measure the increase of the mass of cup. Obtain the mass increases per unit hour of consequtive two weighings respectively, continue this test until it will become constant within 5 %.

The time interval of the weighings shall be 24, 48 or 96 h and its increase in mass shall be at least not less than 5 mg.

Moreover, it is necessary to complete the test before the moisture absorbent put in the cup has absorbed a moisture of 10 % to its mass.

(12) Where the water vapour transmission rate of sample is small or where the sample has a hygroscopic property, produce not less than two blank cups without the moisture absorbent by the same operation, add this to the test specimens to conduct the test similarly and it is desirable to correct the increased mass of test specimen at each time interval by the mean value of the mass change of the blank cup.

Notes (1) The temperature of melted sealing wax shall not be the temperature likely to impair the measurement such as the part corresponding to the water vapour transmission area will melt or shrink.

(2) Where the test piece includes materials such as paper, paper-boards cellophane and the like has been exposed to the other direction, the use of cover is required.

7. Calculation

Obtain the water vapour transmission rate from the following formula on each test specimen and round off it to two significant figures as specified in JIS Z 8401.

$$\text{Water vapour transmission rate } (g/m^2 \cdot 24h) = \frac{240 \times m}{t \cdot s}$$

where s: area of the water vapour transmission (cm^2)

t: total of time intervals between the last two weighings (h)

m: total of increased masses between the last two weighings (mg)

8. Report

Report the test results on the mean value, the minimum value and the maximum value, the number of measured values and the temperature and humidity conditions (the classification of A and B) of the used atmosphere as the water vapour transmission rate ($g/m^2 \cdot 24$ h) according to JIS Z 0208 and append particulars if there are the following items:

(1) The distinction of direction of the test piece when fitted to the cup.

(2) Where the test has been made after having treated the test piece preliminarily.

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Edition 9

Japanese Text

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